

**TECHNOLOGY ASSESSMENT
AND RESEARCH BRANCH**

**TEST PROTOCOL
14-35-30551**

**SUGGESTED TEST PROTOCOL
FOR THE EVALUATION OF
OIL SPILL SKIMMERS
FOR THE OCS
FINAL**

February 1992

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**Prepared under Contract Number:
14-35-30551**

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ACKNOWLEDGEMENTS

The development of this Suggested Test Protocol for the Evaluation of Oil Spill Skimmers for the OCS was funded by the Department of the Interior, Minerals Management Service (MMS). Certain individuals that contributed significantly with their comments, technical evaluations and guidance were Mr. Ed Tennyson, Mr. Larry Hannon and Mr. Joseph Mullin of MMS, Technology Assessment and Research Branch.

INTRODUCTION

This Suggested Skimmer Test Protocol is based in part on methods previously developed to test oil spill booms. Between 1984 and 1988, the Ohmsett National Oil Spill Test Facility Interagency Technical Committee (OITC) sponsored the development of the Test Protocol for the Evaluation of Oil-Spill Containment Booms.¹ The OITC member agencies were:

- Department of the Interior, Minerals Management Service (MMS)
- U.S. Environmental Protection Agency (EPA)
- U.S. Coast Guard
- U.S. Navy
- Environment Canada

The major purpose of the protocol is to provide, through a series of tests and simulation modeling, a way to test skimmer effectiveness in the open ocean without having to spill oil. This suggested test protocol for oil spill skimmers is based on the same premise as the boom protocol. The boom protocol uses the principle of super-position to determine the effectiveness of a boom in a random sea. First, the effect a simple sinusoidal wave has on a boom's ability to contain oil and its ability to conform to waves is measured in a wave tank using oil. Several different frequency waves are used. Extrapolations are drawn to include longer period and greater amplitude waves than are possible in the tank. A model is created relating wave conformance and oil containment based on combined wave patterns.

Using the same pressure-defined depth measurement system as used in the boom protocol to determine skimmer wave conformance in open water testing, the response model can be used to calculate the oil recovery (or loss) the device will experience. This is accomplished by using Fourier analysis to determine the component frequencies and wave heights of the irregular sea spectrum and then recreating that sea spectrum with the oil loss model developed from tank testing. It is more difficult to develop a single protocol for skimmer testing than it is for booms because there is a greater variety of operating principles for skimmers. In addition, many skimmers have more than one operating mode. Therefore, the full spectrum of skimmer testing inherently includes parameters that are not common to all skimmers. To address this issue, the test protocol assumes that whatever type of skimmer is being tested, the operating settings are optimum.

Additional supporting information concerning instrumentation calibration, physical and chemical analyses, data processing, and quality assurance are enclosed in the Appendices.

In general, the effectiveness of a skimmer in recovering oil decreases with increasing wave action and increasing current speed. Figures 1, 2 and 3 graphically show the combined effects of harbor chop (HC) waves (compared to calm water), increasing tow speed, oil viscosity and density and slick thickness. The dependent variable is throughput efficiency which is the percentage of oil encountered versus that recovered.

¹U.S. EPA "Draft Test Protocol for the Evaluation of Oil-Spill Containment Booms" by Roy F. Weston, Inc. under Contract No. 68-03-3450, September, 1988.

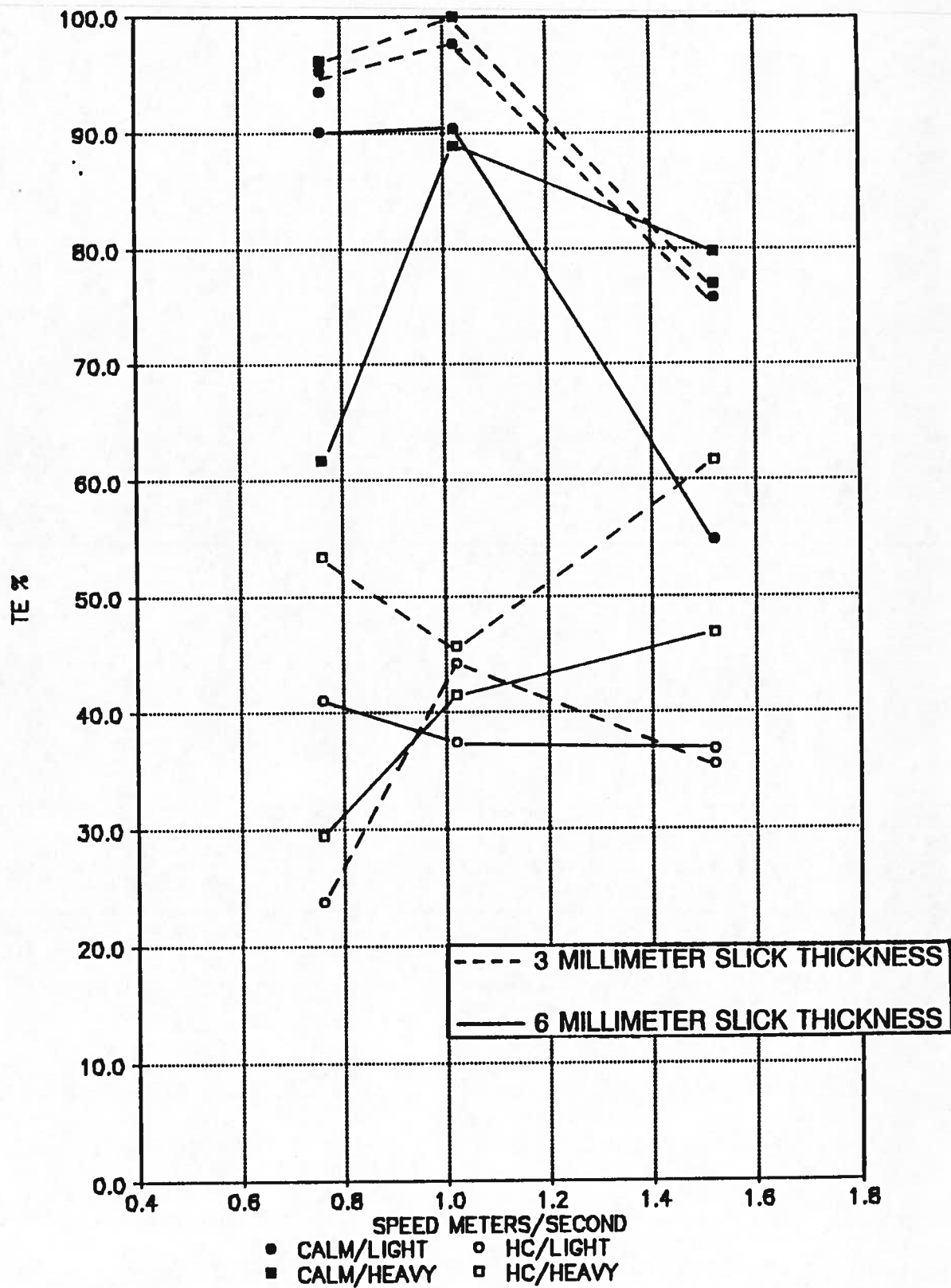


FIGURE 1 - Skimmer L Test, 3 and 6 mm slick thicknesses.

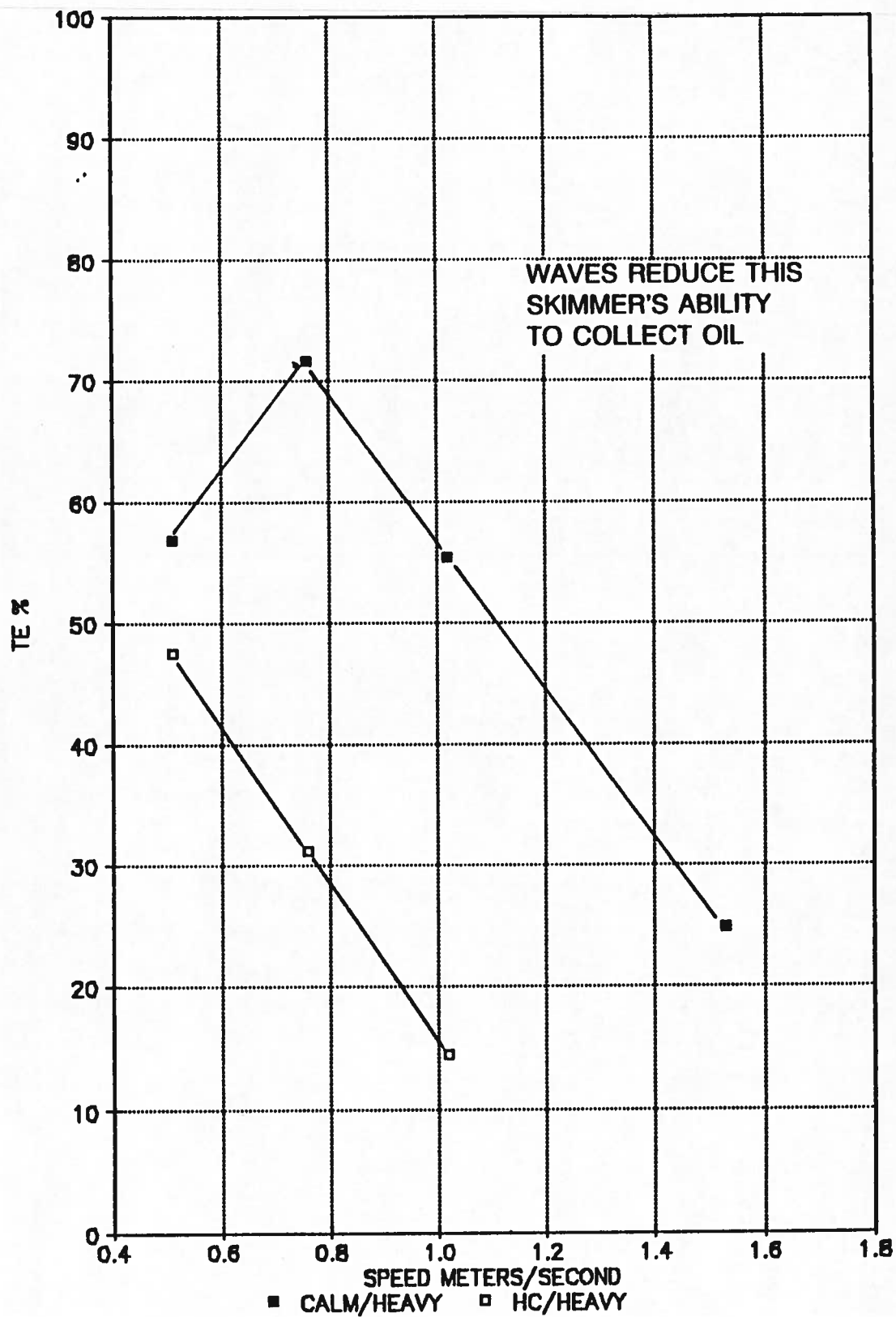


FIGURE 2 - Skimmer M Test, 3 mm slick thickness.

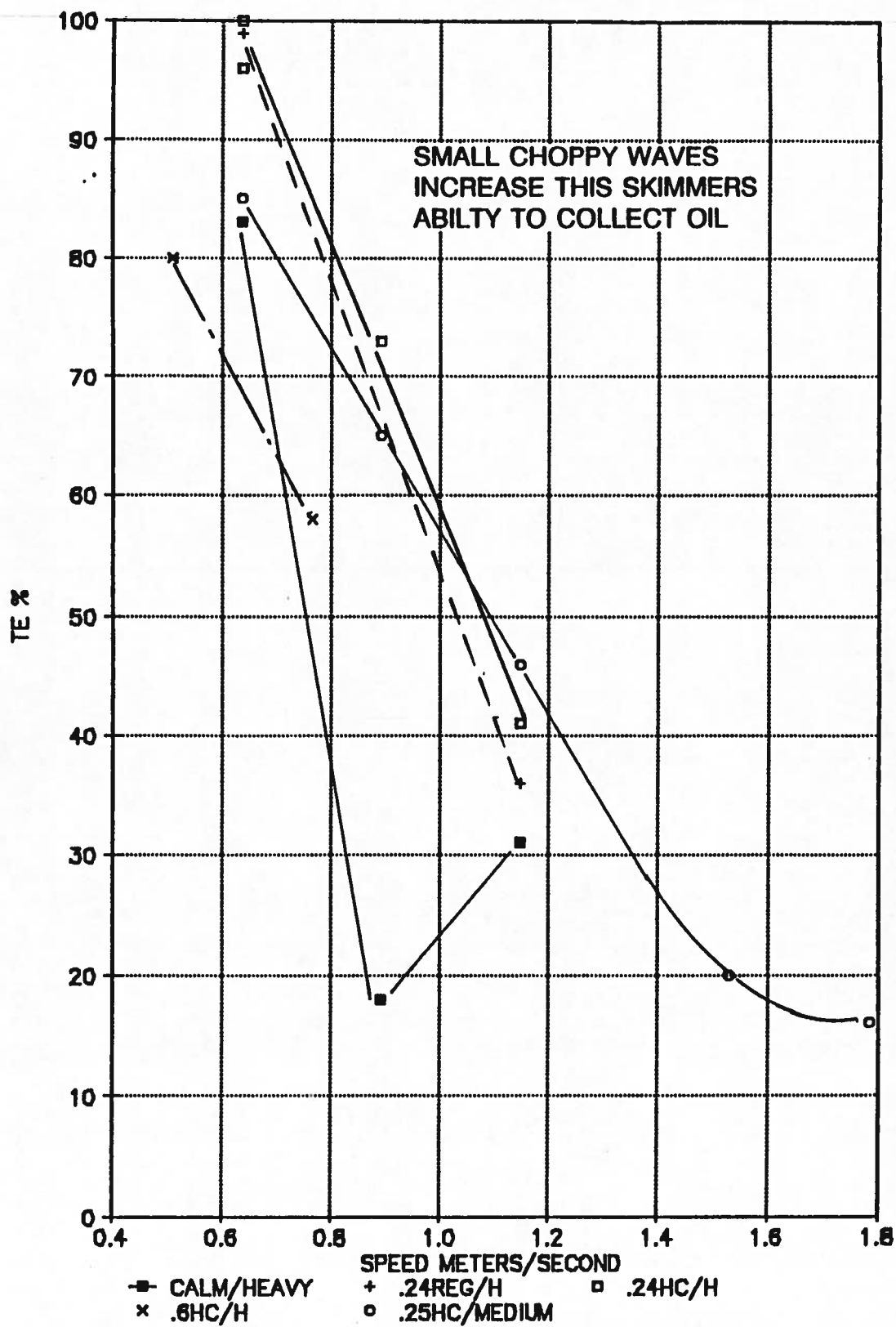


FIGURE 3 - Skimmer F Test, 2 mm slick thickness.

SUGGESTED TEST PROTOCOL FOR THE EVALUATION OF OIL SPILL SKIMMERS FOR THE OCS

1. SCOPE

1.1 Because of environmental restrictions, testing oil spill equipment in waters of the United States is limited to "spills of opportunity" or "dry-runs" where no oil is present. It is likely that such restrictions will continue.

This test protocol is intended for use with American Society for Testing and Materials (ASTM) Methods F 631 and F 808.

1.2 This protocol specifies an approach to test oil spill skimmers for effectiveness in off-shore environments. By measuring skimmer depth over time during standard spills of opportunity or tank tests for oil recovery rate (ORR), throughput efficiency (TE) and recovery efficiency (RE) and measuring skimmer depth over time in an off-shore environment, ORR, TE and RE can be calculated for the offshore.

A skimmer response model is constructed to predict skimmer performance in wave conditions that are anticipated for actual use. The effect of tow speed and wave motion are correlated to ORR, TE, and RE from regular wave tank test data. The reduced performance of these recovery characteristics from calm water values is used to construct model performance in wave spectra as might be encountered in open water. The model of skimmer performance is the added effects of the waves that make up the spectrum and is treated in a similar fashion to vessel response amplitude operators.

2. SUMMARY OF THE METHOD

2.1 Over many years of testing oil skimming devices, it has been well demonstrated that wave conditions and relative current speed are significant factors in oil recovery. See Figures 1, 2 and 3. By measuring the relative depth of a skimmer over time to the water surface in tests at various wave and speed conditions, a

skimmer response model can be constructed. During tank tests, ORR, TE and RE measurements can also be made. A second model can then be constructed linking oil recovery performance to the skimmer wave response.

3. SIGNIFICANCE

3.1 This approach lets investigators predict a skimmer's oil recovery efficiency in wave conditions not attainable in a test tank. It is a recommended approach, not yet proven, based on the success of its application to oil spill booms.

4. APPLICABLE DOCUMENTS

4.1 Test Protocol For the Evaluation of Oil Spill Containment Booms

4.2 ASTM Standard Methods:

F 631 Standard Method for Testing Full Scale Advancing Spill Removal Devices.

F 808 Standard Guide for Collecting Skimmer Performance Data in Uncontrolled Environments.

5. TANK TESTS

5.1 Tests run under controlled and restricted conditions result in data that is used to predict behavior in open water. A test tank where oil can be distributed on water is required to determine the relationship between skimmer draft maintenance and oil recovery efficiency. The test tank must meet dimensional criteria and specific oil handling apparatus is required.

5.2 Apparatus:

- Wave tank.
- Skimmer.
- Skimmer towing equipment or water recirculating pumps.
- Tow speed or current meter.
- Wave meter, electronic output.

- Pressure Sensors.
- Multi-channel analog or digital datalogger.
- Video cameras, underwater and surface.
- Test oil.
- Oil Distribution system.
- Oil collection, skimming system.
- Oil recovery tanks.
- Stratified fluid samplers.
- Sample bottles, 250 milliliters (ml).
- Laboratory centrifuge, 980 G.
- Centrifuge tubes, 100 ml pear shaped.
- Toluene.
- Radio or intercom communication system.
- Anemometer, 10 meters (m) above tank.
- Thermometers, for air and water.
- Hydrometers for oil and tank water.

5.2.1 The tank must have the following characteristics:

Length: For test tanks where the skimmer is moved through stationary water, the test tank must be long enough to allow for a minimum of two consecutive 30-second skimming durations that show equilibrium (equivalent oil recovery). Depending on the pre-test conditions for sumps, hoses and storage tanks and the maximum skimmer speed, acceleration and stopping multiplied by the minimum test duration would be 1.5 minutes.

Width: In order to minimize wall effects, the test tank will be wide enough to provide clearance between the wall and skimmer that equals two and a half times skimmer draft.

Depth: In order to minimize bottom effects, the water depth in the tank must be a minimum of four times the skimmer draft.

Tow Speed Requirements (Water Circulation Requirements): The test facility shall have the ability to vary the relative velocity between the skimmer and the water a maximum of 300 centimeters (cm) per second (6 kt).

Wave Generation: The ability to generate both sinusoidal and harbor chop (random sea)

waves is required.

Oil Distribution: Oil distribution is required to provide a 1, 5 and 25 millimeter (mm) slick thickness across the effective width of the skimmer (100% encounter).

TABLE 1 - OIL SLICK THICKNESS DISTRIBUTION PUMPING FACTORS - GPM/FT WIDTH

DISTRIBUTION SPEED (M/SEC) (FT/MIN)		DESIRED SLICK THICKNESS (MILLIMETERS)		
		1	5	25
0.10	20	0.48	2.41	12.06
0.20	39	0.96	4.82	24.11
0.25	49	1.21	6.03	30.14
0.30	59	1.45	7.23	36.17
0.40	79	1.93	9.65	48.23
0.51	100	2.46	12.30	61.49
0.60	118	2.89	14.47	72.34
0.70	138	3.38	16.88	84.40
0.76	150	3.67	18.33	91.63
0.80	157	3.86	19.29	96.46
0.90	177	4.34	21.70	108.51
1.02	201	4.92	24.60	122.98
1.10	217	5.31	26.53	132.63
1.27	250	6.12	30.62	153.12
1.30	256	6.27	31.35	156.74
1.40	276	6.75	33.76	168.80
1.50	295	7.23	36.17	180.86
1.53	301	7.38	36.89	184.47
1.60	315	7.72	38.58	192.91
1.70	335	8.20	40.99	204.97
1.80	354	8.68	43.41	217.03
1.90	374	9.16	45.82	229.08
2.04	402	9.84	49.19	245.96
3.06	602	14.76	73.79	368.95

5.2.2 **Instrumentation:** The basic instrumentation for skimmer tank testing consists of an oil distribution flowmeter, fluid recovery flowmeter, pressure transmitter(s), tow speed meter and wave meter. Figure 4 depicts a basic test layout showing the relative locations of each instrument. Figures 5 and 6 show pressure transmitter placements for four different skimmer types. The outputs from the instruments are used to measure or calculate the following:

Oil Distribution Flowmeter:
Volume of encountered oil
Oil slick thickness

Fluid Recovery Flowmeter:
 Fluid recovery rate
 Oil recovery rate
 Throughput efficiency

Pressure Transmitter(s)*
 Wave conformance
 Tow Speed Meter
 Tow speed
 Slick thickness with oil distribution

Wave Meter
 Wave profile
 Fourier transformed wave frequency

* Pressure transmitters are electro-mechanical sensors that respond to fluid pressures by passing an electric current proportional to the pressure. This type of measurement system is described as "low impedance loop" and operates in the milliamp range. In contrast to voltage output systems that tend to pick up induced signals in electrically "noisy" areas these transmitters do not.

5.3 Wave conditions for tank testing should include wave heights from 19 to 60 cm and wave periods between 1.5 and 7 seconds.

TABLE 2 - WAVE CONDITIONS
 FOR SKIMMER TESTING

PERIOD (SEC.)	HEIGHT (CM)
7	15
3.5	32
3.5	50
2.5	15
2.5	30
2.5	45
1.7	20

5.4 Tow Speeds for Advancing Skimmers are: 0.25, 0.51, 1.02, 1.27 and 1.53 m/sec. For high speed skimmers (skimmers that function up to 3.06 m/sec), the increments are: 0.25, 0.51, 1.02, 2.04 and 3.06.

5.5 Pretest Setup: Skimmers that are normally towed should be rigged as designed using the tank towing mechanism. Self-

propelled skimmers and vessel of opportunity skimmers, for safety reasons, must also be towed. Rigging for tank testing must allow these devices some freedom to respond to the waves naturally. Consideration must also be made if data is generated towing from the bow when the device is propelled from the stern in actual use. See Figures 4, 5, and 6.

5.6 Tank Testing: Begin tank testing by determining the behavior of the skimmer and the adequacy of the test rig in the wave conditions and over the speed range that will be used for oil recovery tests. Observe the skimmer and monitor the output signal(s) from the pressure transmitter(s) while towing through the following waves: 7 second, 15 cm; 3.5 second, 32 cm; 2.5 second, 42 cm and 1.7 second, 19 cm. The tow speeds will depend on the performance range of the device. For low speed skimmers, they are: 0.25, 1.02 and 1.53 m/sec. and for high speed skimmers, 0.51, 1.53 and 3.06 m/sec.

Calculate the appropriate oil distribution rates to obtain 1,5 and 25 mm slick thicknesses at the encounter width of the skimmer.

Where:

Rate = W x factor in TABLE 1

Example: If the encounter width is 2.0 meters and the test speeds are 0.25, 0.51, 1.02, 1.27 and 1.53 m/sec., pumping rates will be:

TABLE 3 - EXAMPLE OF OIL DISTRIBUTION IN GPM
 FOR TWO METER SKIMMER ENCOUNTER WIDTH

SPEED	THICKNESS (mm)		
	1	5	25
0.25	-	39.6	197
0.51	-	80.7	403
1.02	32.3	161	-
1.27	40.1	200	-
1.53	48.4	242	-

After creating the table, conduct oil recovery tests for the ten speed/oil distribution rates in both calm water and in 3.5 second, 32 cm waves. For each ten tests, conduct a duplicate

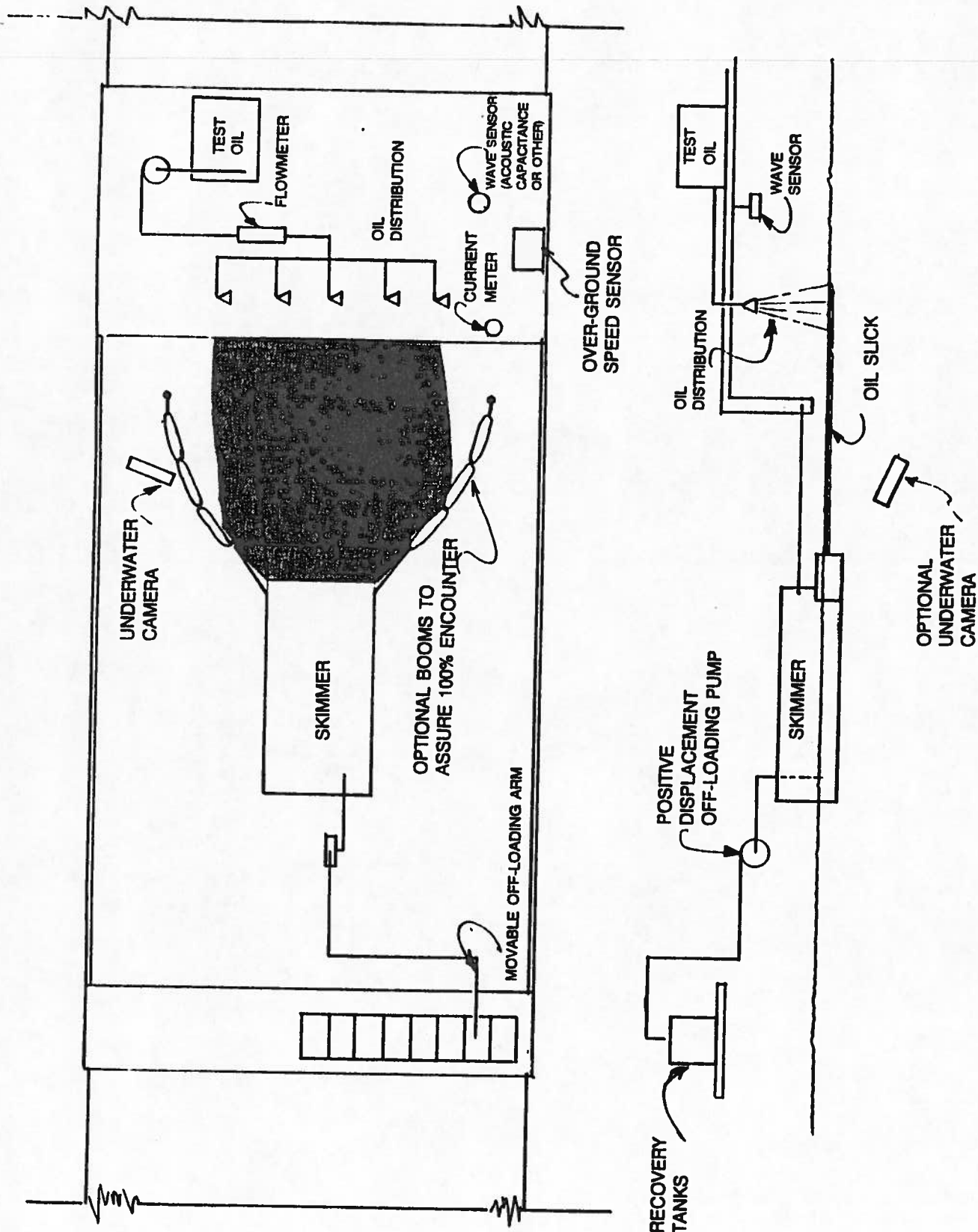
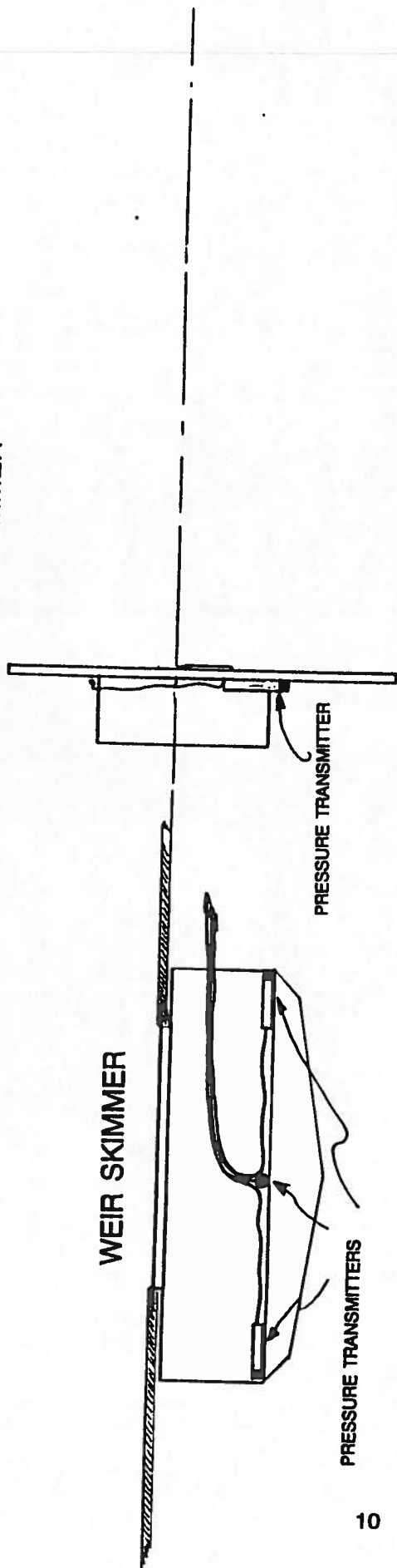


FIGURE 4 - Tank test layout for skimmer testing.

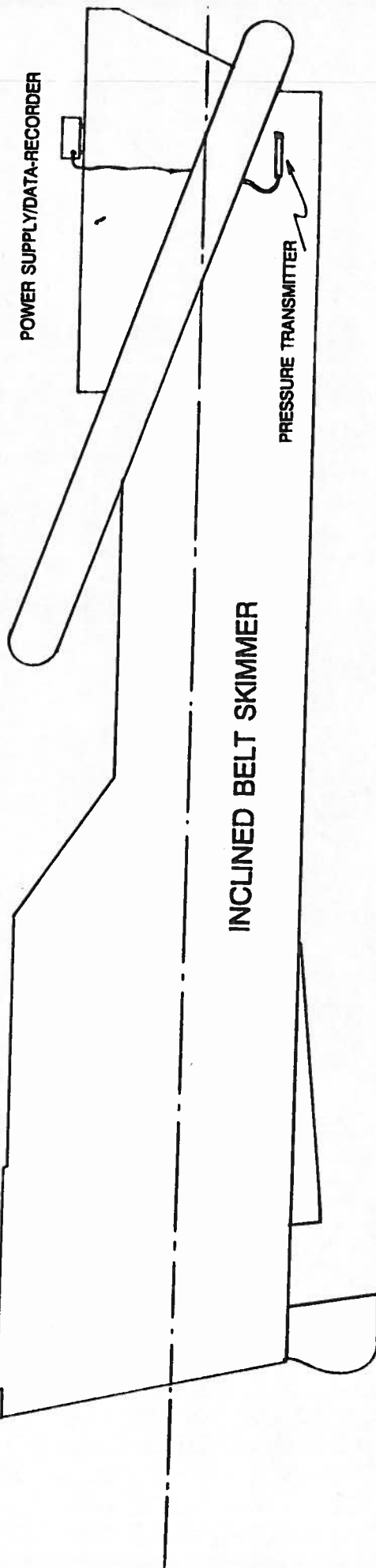
BARRIER WITH BUILT-IN SKIMMER



WEIR SKIMMER

PRESSURE TRANSMITTER

PRESSURE TRANSMITTERS



INCLINED BELT SKIMMER

PRESSURE TRANSMITTER

POWER SUPPLY/DATA-RECORDER

FIGURE 5 - Pressure transmitter placement locations for three skimmer types.

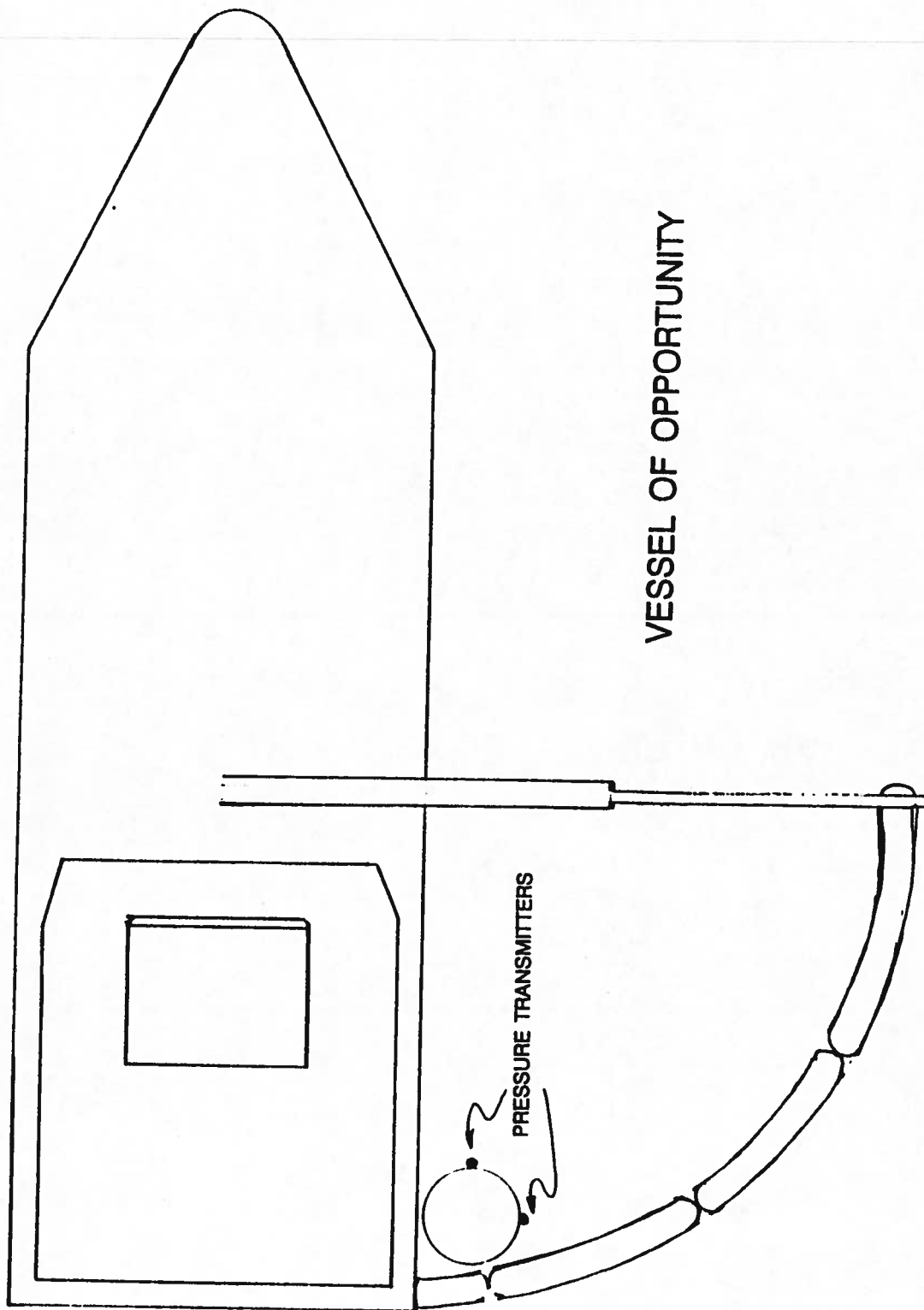


FIGURE 6 - Pressure transmitter location for a skimmer on a vessel of opportunity.

test. A test is five minutes in duration or a series of subtests that total five minutes at the required operating conditions.

At the end of each test, sample the fluid recovered by the skimmer as prescribed in the methods section. (See the following section 5.7 Sampling.) Samples must be taken from containers of known depth/volume characteristics. Samples from irregularly-shaped tanks (such as a skimmer's on-board storage) should be avoided. Sampling must be conducted to evaluate at least two consecutive 30 second periods.

For speed and slick thickness conditions that the skimmer achieves an 50% or better throughput efficiency, conduct tests in 2.5 second, 42 cm and 7 second, 15 cm waves.

5.7 Sampling: In order to meet the requirement of at least two consecutive 30-second periods that have equivalent oil recovery, the skimmed test fluid must be either segregated according to its collection time or sampled from recovery lines during skimming. The fluids recovered by a skimmer are alternately two, one or three phases, making line samples unpredictable. Past experience has shown that using a series of recovery tanks (bypassing on-board storage if necessary) is a satisfactory way to collect recovered fluids for subsequent sampling.

5.8 Open Water Testing: The open water tests consist of monitoring the relative position of the active part of the skimmer with respect to the water surface under existing wave and wind conditions. The maximum specified wave conditions for this test are sea state 4. The test location and schedule should be such that sea state 4 will occur for one or more hours during a 24-hour period.

Pressure transmitters are attached to the skimmer as done before for tank testing.

The skimmer is operated into head seas at the five lower or five upper speeds as done during tank testing. Each speed will be maintained for five minutes.

During each five minute test, pressure transmitter signals will be recorded for subsequent analysis.

After all speeds have been monitored in head seas, the tests are repeated in following seas, again in head seas and yet again in following seas making a total of four tests with five speeds each.

During the open water tests, wave height and period, wind speed and direction and skimmer to water relative current speed is continuously monitored and recorded. The wave buoy or wave staff should be no more than 500 meters from the test area and should not be in the lee of the skimmer or support vessels.

6. CALCULATIONS

6.1 Calculate for tank tests as follows:

Percent Recovery Efficiency (%RE): Percent RE's are calculated from measurements of the fluid recovered during each of the steady state periods and the percent oil recovered. If water is decanted from the recovered fluid prior to sampling, the % RE is equal to:

$$\frac{\text{volume of recovered oil} \times (1 - \% \text{ bs \& w}) \times 100}{\text{total volume of fluid recovered}}$$

Where:

bs = bottom solids and

w = water

Oil Recovery Rate (ORR): ORR's are calculated from the measurements of the oil recovered during each of the steady state periods and the duration of each of the steady state periods in minutes.

Percent Throughput Efficiency (%TE): %TE's are calculated from the measurements of oil recovered during each of the steady state periods and the oil encountered during that same steady state period. It is best to assure a 100% encounter with all the oil distributed to simplify this calculation. (See the optional boom in Figure 4.)

$$\frac{\text{volume of oil recovered (1 - \% bs \& w)}}{\text{distribution pumping rate} \times \text{period duration} \times \%E}$$

Where:

bs = bottom solids

w = water and

%E = the percent encounter expressed as a decimal.

Using Fourier Transforms, determine any periodicities and the corresponding magnitude in the pressure transmitter signals monitored during the steady state durations. If the program supplied with this protocol is used, a minimum of 1024 data points must be analyzed.

Encounter periodicity (T_e): Encounter periodicities are calculated from measured wave periodicities and the forward velocity. If the wave sensor is traveling with the skimmer as in Figure 4, the measured periodicity is the encounter periodicity. If not, then:

$$T_e = \frac{L_w}{V + V_w}$$

Where:

L_w = wave length

V = skimmer speed and

V_w = wave velocity

Wave velocity is equal to $(gL_w/2\pi)^{1/2}$ and L_w is equal to $(T^2g/2\pi)$

Where:

T = the period of the wave and

g = acceleration due to gravity

Calculate, using regressions, functional relationships between wave periodicities and heights with %TE, %RE and ORR. Also, calculate, using regressions, functional relationships between wave periodicities and heights with pressure transmitter signal periodicities and magnitude.

6.2 Open Water Tests: Using Fourier Transforms, determine any periodicities and the corresponding magnitudes in the pressure

transmitter signals monitored during each five minute speed run. See Appendix A, PROTOCOL.BASS

Use the equation above to calculate encounter periodicities.

Calculate, using regressions, functional relationships between wave periodicities and corresponding magnitudes with pressure transmitter signal periodicities and corresponding magnitudes for comparison with tank-derived functional relationships. If the functional relationships between tank waves and pressure transmitter signals are equivalent to the open water relationships, graphically determine %TE, %RE and ORR for the open water conditions.

7. REPORT

7.1 Report the following information (See Standard Data Forms Figures 7 and 8):

Skimmer:

manufacturer
model
type (weir, disk etc.)
encounter width
draft
weight
length overall
length at waterline
beam
freeboard

Testing:

test #
date
time
slick thickness
wave period
encounter period
wave height

Oil:

type
condition (new, weathered etc.)
viscosity
density
surface tension
interfacial tension

Recovered Fluid:

- volume
- free water
- bottom solids and water
- recovery time (duration)
- recovered oil

Performance:

- throughput efficiency
- recovery efficiency
- oil recovery rate

7.2 Report functional relationships between the magnitude spectrum, skimmer speed and ORR, TE and RE.

7.3 Report the calculated ORR, TE and RE for the open water conditions using the tank test-derived functional relationships.

GLOSSARY

BARRIER, BOOM	any floating mechanical device intended to prevent the spread of floating oil, increase the thickness of floating oil, or divert the flow of floating oil.
TEST TANK	a wave tank which can create a relative velocity between a boom and the water surface.
TOW SPEED	the relative speed difference between a barrier and the water in which the barrier is floating. In this protocol "current speed" is equivalent.
PRELOAD	during testing, the quantity of oil distributed in front of and contained by the barrier prior to the onset of oil loss.
BARRIER APEX	the portion of the barrier which is farthest from the barrier tow points when towed in symmetric catenary.
BARRIER DRAFT	the maximum distance below the calm-water surface of any boom segment not part of the towing assembly or connector. (D)
BARRIER FREEBOARD	the vertical height of the barrier above the water line in calm water. (F)
HARBOR CHOP	an irregular condition of the water surface produced by an interference pattern of waves. This is also known as random sea.
BARRIER DEPTH	the perpendicular distance from an imaginary line between barrier tow points to the apex.
SIGNIFICANT HEIGHT	when measuring waves, an average of the highest one half, one third or one tenth are used to characterize the sea surface. The "one third significant wave height" is commonly use.
✓ OIL RECOVERY RATE (ORR)	the volume of oil recovered by the skimmer per unit time.
OIL RECOVERY EFFICIENCY (ORE)	the ratio of the volume of oil recovered to the volume of fluid ^{oil} and water collected.
✓ THROUGHPUT (TE)	the ratio of the volume of oil recovered to the volume of oil encountered by the skimmer.
PRINCIPLE OF SUPERPOSITION	if a physical system is acted on by a number of independent influences, the resultant influence is the sum (vector or algebraic as circumstances dictate) of the individual influences.
FOURIER ANALYSIS	(Harmonic Analyses). Any function which is periodic in the independent variable, no matter how complicated the periodicity, can always be regarded as composed of a series of simple harmonic terms.

APPENDIX A

STANDARD TEST METHODS

APPENDIX A

STANDARD TEST METHODS

The following methods are based on ASTM, EPA and "Standard Methods", American Waste Water Association standards and are used to measure the physical characteristics required for this protocol.

OIL AND GREASE, TOTAL RECOVERABLE (Infrared)

A. SCOPE AND APPLICATION

1. This method include the measurement of Freon extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter.
2. The method is applicable to measurement of most light petroleum fuels, although loss of about half of any gasoline present during the extraction manipulations can be expected.
3. The method covers the range for 0.2 to 1000 mg/l of extractable material.

B. SUMMARY OF METHOD

The sample is acidified to a low pH (less than 2) and extracted with Freon. The oil and grease is determined by comparison of the infrared absorbance of the sample extract with standards.

C. INTERFERENCES

The definition of oil is based on this infrared procedure. The source of the oil and the presence of extraneous matter may influence the material measured and the interpretation of results.

D. COMMENTS

1. The minimum concentration of 5 mg/l for quantifying oils with unknown identity is derived from an EPA method for Oil and Grease.
2. Carbon Tetrachloride is more efficient than Freon 113 for extracting high concentrations (greater than 200 mg/l) of viscous oils (100 centistokes at 100°F) from water dispersions.
3. Freon 113 is less hazardous to exposed laboratory personnel than carbon tetrachloride (1000 ppm TLV vs. 10 ppm TLV) and is therefore preferred in situations where adequate ventilation may be lacking.
4. Carbon tetrachloride is always the solvent of choice when: a) oil appears as a separate layer, or b) the sample exhibits discoloration.
5. Difficult to handle emulsions may form when oil in water concentrations exceed the method's concentration limit (1000 mg/l).

E. DEFINITIONS

1. The definition of grease and oil is based on the procedure used. The source of the oil and/or grease and the presence of extractable non-oily matter will influence the material measure and interpretation of results.

2. An "Unknown Oil" is defined as one for which a representative sample of the oil or grease is not available for preparation of a standard. Examples of unknown oils are the oil and grease in a mixed sewerage or an unidentified oil slick on a surface water.
3. A "Known Oil" is defined as a sample of oil and/or grease that represents the only material of that type used or manufactured in the processes represented by a wastewater.

F. APPARATUS

1. Separatory funnel, 2000 ml, with Teflon stopcock.
2. Infrared spectrophotometer, double beam, recording.
3. Cells, quartz, 10 mm and 100 mm path length.
4. Syringes, 10, 25, 50, 100 microliter capacity.
5. Filter paper, Whatman No. 40, 11 cm.

G. REAGENTS

1. Sulfuric acid, 1:1. Mix equal volumes of concentrated H_2SO_4 and distilled water. (Concentrated hydrochloric acid may be substituted directly for concentrated sulfuric acid for this reagent).
2. Freon 113, b.p. 48°C , 1, 1, 2-trichloro-1, 2, 2-trifluoroethane. Freon 113 is available from E.I. DuPont de Nemours, Inc. and its distributors, in 5-gallon cans. It is best handled by filtering one gallon quantities through paper into glass containers. and maintaining a regular program of solvent blank monitoring.
3. Sodium sulfate, anhydrous crystal.
4. Known oil reference standard. Accurately weigh about 0.05 g of known oil directly into a 100 ml volumetric flask. Add 80 ml Freon and dissolve the oil. If, as in the case of a heavy fuel oil, all the oil does not go into solution, let stand overnight. The next day, filter through paper into another 100 ml volumetric flask and dilute to mark. Treat calculations as if all oil had gone into solution.
5. Unknown oil reference standard (10 ml = 7.69 mg oil). Pipette 15.0 ml n-hexadecane, 15.0 ml isooctane, and 10.0 ml toluene into a 50 ml glass stoppered bottled. Assume the specific gravity of this mixture to be 0.769 and maintain the integrity of the mixture by keeping stoppered except when withdrawing aliquots.

H. PROCEDURE

1. Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 2 ml sulfuric or hydrochloric acid (G.1) to the sample bottle. After mixing the sample, check the pH by touching pH sensitive paper to the cap to ensure that the pH is 2 or lower. Add more acid if necessary.

2. Pour the sample into a separatory funnel.
3. Add 30 ml Freon (G.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate.
4. Filter the solvent layer into a 100 ml volumetric flask through a funnel containing solvent-moistened filter paper.

Note: An emulsion that fails to dissipate can be broken by pouring 1 g sodium sulfate (G.3) into the filter paper cone and draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.

5. Repeat steps H.3 and H.4 twice more with 30 ml portions of fresh solvent, combining all solvent in the volumetric flask.
6. Rinse the tip of the separatory funnel, filter paper and the funnel with a total of 10-20 ml Freon and collect the rinsings in the flask. Dilute the extract to 100 ml and stopper the flask.
7. Select appropriate calibration standards and cell pathlength according to the following table of approximate working ranges:

<u>PATHLENGTH</u>	<u>RANGE</u>
1 cm	4-40 mg/liter
5 cm	0.5-8 mg/liter
10 cm	0.1-4 mg/liter

Prepare calibration standards by pipetting appropriate amounts of the known oil reference standard (G.4) into 100 ml volumetric flasks and diluting to mark with Freon. Alternately, transfer appropriate amounts of the unknown oil reference standard (G.5) using microliter syringes to 100 ml volumetric flasks and diluting to mark with Freon.

Note: Ten microliters of the unknown oil is equivalent to 7.69 mg per 100 ml Freon and 7.69 mg per sample volume.

8. Scan standards and samples from 3200 cm^{-1} to 2700 cm^{-1} with Freon in the reference beam and record the results on absorbance paper. The absorbances of samples and standards are measured by constructing a straight baseline over the range of the scan and measuring the absorbance of the peak maximum at 2930 cm^{-1} and subtracting the baseline absorbance at that point. If the absorbance exceeds 0.8 for a sample, select a short pathlength or dilute as required.

Note: Caution must be exercised in the selection of the 2930 cm^{-1} peak as it may not always be the largest peak in the range of the scan. For an example of a typical oil spectrum and baseline construction, see Gruenfeld (3).

9. Use a calibration plot of absorbance vs. mg oil prepared from the standards to determine the mg oil in the sample solution.

I CALCULATION

$\text{mg/l total oil and grease} = \text{RXD/V}$

where:

R = oil in solution, determined from calibration plot in milligrams.

D = extract dilution factor, if used.

V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition, if necessary in liters.

J. PRECISION AND ACCURACY

By this method, a single laboratory determined the oil and grease level in sewerage to be 17.5 mg/l. When 1 liter portions of the sewerage were dosed with 14.0 mg of a mixture of #2 fuel and Wesson oil, the recovery was 99% with a standard deviation of 1.4 mg. See Quality Assurance section.

K. REFERENCES

1. Standard Methods for the Examination of Water and Wastewater, 13th Edition, p. 254, Method 137, (1971).
2. American Petroleum Institute, "Manual on Disposal of Refinery Wastes", Vol. IV, Method 733-58 (1958).
3. Gruenfeld, M., "Extraction of Dispersed Oils from Water for Quantitative Analysis by Infrared Spectroscopy", Environ. Sc. Technology 7,636 (1973).
4. Methods for Chemical Analysis of Water and Wastes, U.S. EPA, 1973, pp. 232-235.

SPECIFIC GRAVITY

A. SCOPE AND APPLICATION

1. This method covers the laboratory determination, using a glass hydrometer, of the density, specific gravity, or API gravity of crude petroleum and non-petroleum products normally handled as liquids, and having a Reid vapor pressure (ASTM Method D323, Test for Vapor Pressure of Petroleum Products (Reid Method) of 26 lbs. or less. Values are measured on a hydrometer at convenient temperatures, readings of density being reduced to 15°C, and readings of specific gravity and API gravity to 60°F, by means of these same tables, values determined in any one of the three systems of measurements are convertible to equivalent values in either of the other two so that measurements may be made in the units of local convenience.
2. The hydrometer method is most suitable for determining the density, specific gravity or API gravity of mobile, transparent liquids. It can also be used for viscous oils by allowing sufficient time for the hydrometer to reach equilibrium, or for opaque oils by employing a suitable meniscus correction.
3. When used in connection with bulk oil measurements, volume correction errors are minimized by observing the hydrometer reading at a temperature close to that of the bulk oil temperature.

B. SUMMARY OF METHOD

The sample is brought to the prescribed temperature and transferred to a cylinder at approximately the same temperature. The appropriate hydrometer is lowered into the sample and allowed to settle. After temperature equilibrium has been reached, the hydrometer scale is read and the temperature of the sample is noted. If necessary, the cylinder and its contents are placed in a constant-temperature bath to avoid excessive temperature variation during the test.

C. COMMENTS

1. The density, specific gravity or API gravity by the hydrometer method is most accurate at or near the reference temperature of 15°C or 60°F.
2. When the hydrometer value is to be used to select multipliers for correcting volumes to standard temperatures, the hydrometer reading should be made preferable at a temperature within $\pm 3^\circ\text{C}$ ($\pm 5^\circ\text{F}$) of the temperature at which the bulk volume of the oil was measured.

D. DEFINITIONS

1. Density - for the purpose of this method, the mass (weight in volume) of liquid per unit volume at 15°C. When reporting results, explicitly state the density in units of mass (kilograms) and volume (liters) together with the standard reference temperature, e.g. kilograms per liter at 15°C.
2. Specific Gravity - for the purpose of this method, the ratio of the mass of a given volume of liquid at 60°F to the mass of an equal volume of pure water at the same temperature. When stating results explicitly report the standard reference temperature, e.g. specific gravity 60/60°F.

3. API gravity - a special function of specific gravity 60/60°F represented by:

$$\text{API gravity, deg.} = (14.5/\text{sp gr } 60/60^\circ\text{F}) - 131.5$$

No statement of reference temperature is required, since 60°F is included in the definition.

4. Observed values - since all hydrometers are calibrated to read corrected at a specified reference temperature, values observed at other temperatures are only hydrometer readings and not density, specific gravity or API gravity at that other temperature.

E. APPARATUS

1. Hydrometers, glass, graduated in units of density, specific gravity or API gravity as required, conforming to ASTM specifications or specifications of the British Standards Institution.
2. Thermometers having a range from -2 to 40° centigrade and conforming to specifications of the ASTM or the Institute of Petroleum.
3. Hydrometer Cylinder, clear glass, plastic or metal. For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the hydrometer floats in the sample with at least 25 mm clearance between the bottom of the hydrometer and the bottom of the cylinder.

Note: Hydrometer cylinders constructed of plastic materials shall be resistant to discoloration or attack by oil samples and must not become opaque under prolonged exposure to sunlight and oil samples.

4. Constant Temperature Bath, for use when the nature of the sample requires a test temperature much above or below room temperature.

Note: The user should ascertain that the instruments used for this test conform to the requirements set out above with respect to materials, dimensions and scale errors. In cases where the instrument is provided with a calibration certificate issued by a recognized standardizing body, the instrument is classed as "certified" and the appropriate corrections listed shall be applied to the observed readings. Instruments which satisfy the requirements of this test method, but are not provided with a recognized calibration certificate are classified as "uncertified".

F. PROCEDURE

1. Adjust the temperature of the sample according to the indications given above. Bring the hydrometer cylinder and thermometer to approximately the same temperature as the sample to be tested.

Note: When testing completely opaque samples, metal hydrometer cylinders may be used. When such cylinders are used, accurate reading of the hydrometer can only be assured if the level of the sample is within 5 mm of the top of the cylinder.

2. Transfer the sample to a clean hydrometer cylinder without splashing to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower boiling constituents of the more volatile samples to the cylinder by water displacement or by siphoning. Remove any air bubbles formed, after they have collected on the surface of the sample by touching them with a piece of clean filter paper before inserting the hydrometer.

Note: Highly volatile samples containing alcohols or other water-soluble material should always be transferred by siphoning.

3. Place the cylinder containing the sample in a vertical position in a location free from air currents. Ensure that the temperature of the sample does not change appreciably during the time necessary to complete the test; during this period, the temperature of the surrounding medium should not change more than 2°C (50°F). When testing at temperatures much above or below room temperature, a constant-temperature bath may be necessary to avoid excessive temperature changes.
4. Lower the hydrometer gently into the sample. Take care to avoid wetting the stem above the level to which it will be immersed in the liquid. Continuously stir the sample with the thermometer, taking care the mercury thread is kept fully immersed and that the stem of the hydrometer is not wetted above the immersion level. As soon as a steady reading is obtained, record the temperature of the sample to the nearest 0.25°C (0.5°F) and then remove the thermometer.
5. Depress the hydrometer about two scale divisions into the liquid and then release it. The remainder of the stem of the hydrometer which is above the level of the liquid must be kept dry since unnecessary liquid on the stem affects the reading obtained. With samples of low viscosity, impart a slight spin to the hydrometer on releasing to assist in bringing it to rest, floating freely away from the walls of the cylinder. Allow sufficient time for the hydrometer to come to rest and for all air bubbles to come to the surface. This is particularly necessary in the case of more viscous samples.
6. When the hydrometer has come to rest, floating freely away from the walls of the cylinder, estimate the hydrometer scale reading to the nearest 0.001 sp gr or density or 0.05 deg API. The correct hydrometer is that point on the hydrometer scale at which the principal surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.
7. With an opaque liquid, take a reading by observing with the eye slightly above the plane of the surface of the liquid, the point on the hydrometer scale to which the sample rises. This reading, at the top of the meniscus, requires correction since hydrometers are calibrated to be read at the principal surface of the liquid. The correction for the particular hydrometer in use may be determined by observing the maximum height above the principal surface of the liquid to which oil rises on the hydrometer scale when the hydrometer in question is immersed in a transparent oil having a surface tension similar to that of the sample under test.
8. Immediately after observing the hydrometer scale value, again cautiously stir the sample with the thermometer keeping the mercury thread fully immersed. Record the temperature of the sample to the nearest 0.2°C (0.5°). Should this temperature differ from the previous reading by more than 0.2°C (0.5°), repeat the hydrometer and then thermometer observations until the temperature becomes stable within 0.5°C (1°F).

Note: After use at a temperature higher than 38°C (100°F), allow all hydrometers of the lead shot in wax type to drain and cool in a vertical position.

G. CALCULATIONS AND REPORT

1. Apply any relevant corrections to the observed thermometer reading (for scale or bulb) and to the hydrometer reading (scale). For opaque samples, make the appropriate correction to the observed hydrometer reading as given in F.7. Record to the nearest 0.0001 density or specific gravity of 0.1 deg API the final corrected hydrometer scale reading. After application of any relevant corrections, record to the nearest 0.5°C or 1°F, the mean of the temperature values observed immediately before and after the final hydrometer reading.

Note: Hydrometer readings at temperatures other than calibration temperatures (15°C or 60°F) should not be considered as more than scale readings.

2. To convert corrected values from G.1 to standard temperature, use the following referenced tables from the The American Petroleum Institute's Petroleum Measurement Tables.
3. When a density-scaled hydrometer has been employed, use Table 53 to obtain density at 15°C.
4. When a specific gravity hydrometer has been employed, use Table 23 to obtain Specific Gravity 60/60°F.
5. When an API gravity-scaled hydrometer has been employed, use Table 5 to obtain the gravity in API degrees.
6. When a value is obtained with a hydrometer scaled in one of the units described herein and a result is required in one of the other units, make the conversion by one of the appropriate tables given in the Petroleum Measurement Tables. For conversion from density at 15°C, use Table 51; from specific gravity 60/60°F, use Table 21; from API gravity, use Table 3.
7. Report the final value as density in kilograms per liter at 15°C, or as specific gravity at 60/60°F, or as gravity in degrees API, as applicable.

H. PRECISION AND ACCURACY

1. The following criteria should be used for judging the acceptability of results.
2. Repeatability: Duplicate results by the same operator should be considered suspect if the results differ by more than the following amounts:

Product	Temperature Range	Units	Repeatability
Transparent	-2 to 24.5°C	Density	0.0005
Nonviscous	29 to 76°F	Specific gravity	0.0005
	42 to 78°	API gravity	0.1
Opaque	-2 to 24.5°	Density	0.0006
	29 to 76°F	Specific gravity	0.0006
	42 to 78 °F	API gravity	0.2

3. **Reproducibility:** The results submitted by each of two laboratories should not be considered suspect unless the results differ by more than the following amounts:

Product	Temperature Range	Units	Repeatability
Transparent	-2 to 24.5°C	Density	0.0012
Nonviscous	29 to 76°F	Specific gravity	0.0012
	42 to 78°F	API gravity	0.3
Opaque	-2 to 24.5°C	Density	0.0015
	29 to 76°F	Specific gravity	0.0015
	42 to 78°F	API gravity	0.5

4. For very viscous products, or when the conditions given in H.2 and H.3 are not complied with, no specific variations can be given.

I. REFERENCES

1. ASTM D287-67
2. ASTM D1298-67

SURFACE AND INTERFACIAL TENSION

A. SCOPE AND APPLICATION

This method covers the measurement under nonequilibrium conditions of the interfacial tension of mineral oils against water, which has been shown by practice to give a reliable indication of the presence of hydrophilic compounds.

B. SUMMARY OF METHOD

Interfacial tension is determined by measuring the force necessary to detach a planar ring of platinum wire from the surface of the liquid of higher surface tension, that is upward from the water-oil interface. To calculate the interfacial tension, the force so measured is corrected by an empirically-determined factor which depends upon the force applied, the densities of both oil and water, and the dimensions of the ring. Measurements are made under rigidly standardized nonequilibrium conditions in which the measurement is completed 1 minute after formation of the interface.

C. APPARATUS

1. Tensiometer with a torsion wire to apply the force to lift the ring; the torsion wire is attached to a scale graduated in dynes/cm.
2. Ring of fine platinum wire in a nearly true circle of 4 or 6 cm circumference welded to a suitable stirrup made of the same wire. It is necessary to know, to three significant figures, the circumference of each ring, and the ratio of the diameter of the ring to the diameter of the wire of which it is made.
3. Sample container: glass beaker or cylindrical vessel having a minimum diameter of 45 mm.

D. SAMPLING AND STORAGE

Filter aqueous samples through a 150 mm diameter, unwashed filter paper of medium porosity.

E. PROCEDURE

1. Clean all glassware by removing any residual oil with Toluene, soap and water, followed by several washes with Freon 113. Rinse thoroughly with tap water and then with distilled water. Unless it is to be used immediately, drain the sample container in an inverted position over a clean cloth.
2. Clean the platinum ring by rinsing it in Toluene and Freon 113. Then heat the ring in the oxidizing portion of a gas flame until orange vapors are no longer visible.
3. Calibrate the tensiometer against known weights and adjust its zero point according to the procedure of its manufacturer. Make certain that all portions of the ring are in the same horizontal plane.

4. Introduce 50 to 75 ml of tank water at a temperature of $25 \pm 1^\circ\text{C}$ to a freshly-cleaned sample container and place it on the adjustable platform of the tensiometer.
5. Slowly lower the platform, increasing the torque of the ring system by maintaining the torsion arm in the zero position. As the film of water adhering to the ring approaches the breaking point, proceed slowly with adjustment to assure that the moving system will be in the zero position when rupture occurs. Using the scale reading at which this occurs, calculate the tension of the water sample as described below using the value of 0.997 for difference of density of water and air ($D-d$); a value of 71 to 72 dynes/cm should be obtained. When low values are found, possibly due to improper adjustment of the tensiometer or improperly-cleaned apparatus, make readjustments; clean the sample container with hot chromic acid cleaning solution, rinse and repeat the measurement. If a low value is still obtained, further purify the distilled water (for instance, by redistilling from an alkaline solution of potassium permanganate).
6. Return the tensiometer scale to zero and raise the adjustable platform until the ring is immersed to a depth of about 5 mm in the distilled water. Pour the filtered oil, previously brought to a temperature of $25 \pm 1^\circ\text{C}$ on the water to a depth of about 10 mm. Take care that the ring does not touch the oil-water interface. If a ring with a short stirrup is used, keep the oil level below the top of the stirrup to prevent bridging. If this is not possible, break the bridge with a suitable clean, sharp instrument as soon as possible after withdrawing the stirrup from the oil.
7. Allow the oil-water interface to age for 30 ± 1 s, then slowly lower the platform, increasing the torque of the ring system by maintaining the torsion arm in the zero position. As the water adhering to the ring approaches the breaking point, proceed slowly with adjustment to assure that the moving parts will be in zero position when rupture occurs. Time these operations so that, as nearly as possible 30 s are required to draw the ring through the interface. Proceed very slowly as the breaking point is approached, since the break is usually sluggish and too rapid movement may result in a high reading. Complete the entire operation, from the time of pouring the oil into the sample container until the film ruptures, in about 1 min. Record the scale reading at which the ring breaks free from the interface.

F. CALCULATION

1. Calculate the interfacial tension of the sample by means of the following equations:

$$\text{Interfacial tension, dynes/cm} = P_c F$$

where:

P = scale reading when film ruptures, dynes/cm and

F = factor converting scale reading in dynes/cm to interfacial tension obtained as described in 6.2.

2. Using the value of diameter ratio, R/r , specified by the manufacturer for the rings used, prepare a graph of correction factors, F , by means of the following equation; the graph should cover even increment of $P/D-d$ from 0 to 800 and should give correction factors to three digits:

$$F = 0.7250 - (0.014520/C^2(D-d)) + 0.4534 - (1.679/Rr)$$

where P = scale reading, dynes/cm
 C = circumference of ring, cm
 D = density of water at 25°C, g/ml
 d = density of sample at 25°C, g/ml
 R = radius of ring, cm and
 r = radius of wire of ring, cm.

G. PRECISION AND ACCURACY

See Quality Assurance Section

H. REFERENCES

ASTM D971-50.

FISHER SURFACE TENSIONOMAT

A. CALIBRATION

The calibration of the torsion wire, and hence of the Surface Tensionomat, has been carefully tested at the factory but should be checked before use and adjusted if necessary. The calibration is carried out so that the dial will read directly in dynes per centimeter.

Weigh a piece of paper (approximately 600 mg) on the analytical balance and record the weight below.

Place the known mass for calibration on the paper platform (600 milligrams is quite suitable and simplifies calculations). Release the torsion arm. Turn the knob on the right side of the case in a counter clockwise direction until the index and its image are exactly in line with the reference line of the mirror. Record the dial reading to the nearest 1/10 scale division (by use of the vernier).

It is now necessary to determine the accuracy of the calibration from the reading obtained. The apparent surface tension, S , is given as follows:

$$S = Mg/2L$$

where:

M = the weight expressed in grams = _____

g = acceleration of gravity expressed in $\text{cm/sec}^2 = 98.7 \text{ cm/sec}^2$

L = mean circumference of the ring in centimeters = _____

S = dial reading = apparent surface tension in dynes/cm = _____

For example, suppose that a 600 milligram weight was used. The circumference of the ring is 6.00 cm and the value for g is 980 cm/sec^2 . Then we find that:

$$S = Mg/2L \quad (0.6 \times 980)/(2 \times 6) = 49.00 \text{ dynes/cm}$$

If the dial reading differs from the calculated value, then the effective length of the torsion arm must be adjusted until these two values do agree. This adjustment is accomplished by turning the knurled knob at the left end of the lever so as to move the hanger hook. If the recorded dial reading is greater than the calculated value, move the hook to shorten the effective length of the arm. Conversely, if the dial reading is less than the calculated value, move the hook to lengthen the effective length of the arm. Repeat the calibration procedure until the dial reading and calculated value agree. The dial will read directly in dynes/cm.

:

VISCOSITY-BROOKFIELD

A. SCOPE AND APPLICATION

This method covers procedures for the empirical measurement of Kinematic Viscosity by use of a Brookfield Viscosimeter.

B. SUMMARY OF METHOD

Viscosity is measure by amount of torque exerted by the fluid against a rotating spindle immersed in the fluid. A torsion wire is connected to a dial calibrated in centipoise (cps).

C. COMMENTS

Correct spindle size is important as each spindle is applicable only in a limited viscosity range.

D. APPARATUS

1. Spindles - various sizes and shapes for various viscosity fluids.
2. Brookfield Model LV viscosimeter.

E. REAGENTS

Calibration fluids (Silicone oils).

F. PREPARATION AND CALIBRATION

1. Periodic calibration is needed for maintaining instrument performance. Calibration fluids are available in several viscosities.
2. These instruments are calibrated to Bureau of Standards values on the basis of immersion in an infinite body with the spindle guard attached. They are accurate to within 1% of full scale when centered in any container over 2 3/4" in diameter. Using the viscosimeters in small containers will reduce the effective range of measurement provided by the #1 and #2 spindles. The Calibration of the #3 and #4 spindles will remain the same regardless of the size of the container used, as long as the guard is attached.
3. The #4 LV spindle has a reduced section in its shaft rather than the groove found on the other spindles. The spindle should be immersed in the fluid to the approximate midpoint of this section.
4. Readings obtained in small containers and/or without the guard can be used only for comparative purposes unless definite correction factors are used with each spindle and with each container. The booklet, "Solutions to Sticky Problems" outlines the procedure to be followed in the establishment of these factors.

5. At 60 rpm, air resistance to rotation has a certain effect on the LV pointer. The values obtained at this speed, with any spindle, should be reduced by 0.4 on the 100 scale (or 2.0 on the 500 scale) before using the Factor Finder provided with the instrument.
6. A condition of turbulent flow is created by the #1 spindle when rotating at 60 rpm in materials having viscosities less than 15 cps. If measurements of absolute accuracy are needed in this region, it is suggested that the U.L. Adapter accessory be used.

G. PROCEDURE

1. Attach spindle to lower shaft. It is best to lift the shaft slightly while it is held firmly with one hand while screwing the spindle on with the other. Care should be taken to avoid putting side thrust on the shaft to protect its alignment.
2. Insert spindle in the test material until the fluid's level is at the immersion groove cut in the spindle's shaft. With a disc-type spindle it is sometimes necessary to tilt the instrument slightly while immersing to avoid trapping air bubbles on its surface. (You may find it more convenient to immerse the spindle in this fashion before attaching it to the Viscometer.) Care should be taken to not hit the spindle against the sides of the fluid container while it is attached to the viscometer, since this too can damage the shaft alignment.
3. Level the viscometer; the bubble level on all models will be of help in this respect.
4. Depress the clutch and turn on the viscometer's motor. following the procedure of having the clutch depressed at this point will prevent unnecessary wear. Release the clutch and allow the dial to rotate until the pointer stabilizes at a fixed position on the dial. The time required for stabilization will depend on the speed at which the spindle rotates; at speeds above 4 rpm, this will generally be about 20-30 seconds, while at lower speeds, it may take the time required for one revolution of the dial. It is possible to observe the pointer's position and stability at low speeds while the dial rotates, but at higher speeds it will be necessary to depress the clutch and snap the motor switch to stop the instrument with the pointer in view. Very little practice is needed to stop the dial at the right point.
5. If check readings are required, start the Viscometer with the clutch still depressed, holding the original reading and then release. This will speed up readings by reducing oscillation of the pointer. If pointer does not stabilize, the material may either thixotropic or its temperature may not be constant. Having the spindle at the temperature of the test material will eliminate the latter possibility.

H. CALCULATIONS

1. Readings obtained from observation of the viscometer dial need to be multiplied by a calibration factor. Each spindle and rotation speed have a different factor. Factors are found using the chart accompanying the viscometer.
2. Results are reported as centipoise or may be converted to other viscosity units.

I. PRECISION AND ACCURACY

See Quality Assurance section

J. REFERENCES

- 1. Operating manuals of Brookfield Instrument Company.**
- 2. ASTM 341-77**
- 3. ASTM D2161-74**

**WATER AND SEDIMENT IN PETROLEUM - CENTRIFUGE
(BOTTOM SOLIDS AND WATER)**

A. SCOPE AND APPLICATION

This method is applicable to oils, fuels and crude oil. The method involves selection of a number of factors such as type and amount of demulsifier, temperature of the sample during testing and the duration of centrifuging. Crudes containing asphaltenes require an aromatic solvent such as toluene. Waxy crudes require that test samples be heated to higher temperatures, emulsions present in some oils may call for the use of demulsifiers and high viscosities and finely divided suspensions often necessitate longer than normal centrifuging times.

B. SUMMARY OF METHOD

A sample is mixed with an appropriate solvent and a demulsifier if needed and is rotated in a centrifuge for a time. The amount of water and sediment is then measured and the percentages calculated from the amount of sample used.

C. APPARATUS

1. Centrifuge - capable of whirling two or more filled centrifuge tubes at a speed that can be controlled to give a relative centrifugal force (rcf) of between 500 and 800 at the tip of the tubes. The revolving head, trunion rings and trunion cups, including the cushions shall be constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs. Calculate the speed of the rotating head as follows:

$$\text{rpm} = 265 \times \text{rcf} \times d$$

where:

rcf = relative centrifugal force, and

d = diameter of swing in inches measured between tips of opposite tubes when in rotation position.

2. Centrifuge tubes - 100 ml, pear-shaped, bottom solids and water annealed glass tubes (such as VWR Scientific brand).

D. REAGENTS

1. The following solvents and demulsifiers have been reported as satisfactory for field testing.

Solvents

Stoddard solvent or other
naphtha solvents with
low aromatic content
Toluene
Xylene
Kerosene
White Gasoline

Demulsifiers

Commercial crude oil demulsifiers

Phenol
Nitrogen gases
Naphthenic acids

2. Water-saturated toluene is the preferred solvent. Other solvents may be used when the clients involved are satisfied that they will provide equivalent results. The majority of solvents will dissolve varying amounts of water. Water saturation at either centrifuging temperature or at test temperature is imperative, but the solvent shall be free of suspended water. This may be accomplished by the addition of 2 ml of water to a centrifuge tube filled with solvent which is then placed in a bath maintained at centrifuge grindout temperature. Shaking will aid in saturation, but adequate settling time or centrifuging is necessary to ensure that the solvent is free of suspended water before use. Kerosene and Stoddard Solvent dissolve a negligible amount of water and do not require water saturation. Gasoline containing tetraethyl lead or de-icer compounds should never be used. Unleaded motor fuels generally contain aromatic compounds and require water saturation before use.
3. The use of a demulsifier is generally beneficial and may be used where comparative tests demonstrate a need. The type and concentration is not limited provided that the demulsifier itself does not contribute to the water and sediment.

E. SAMPLING AND STORAGE

The sample must be thoroughly representative of the material in question and the portion used shall be thoroughly representative of the sample. This requires the addition of a suitable amount (2-3 drops) of surfactant (Igepal 540) and the vigorous agitation of a sample before transfer to a centrifuge tube. Prior to the transfer of any sample to centrifuge tube, vigorous shaking must be repeated.

F. PROCEDURE

1. Fill the centrifuge (one or two tubes may be used) to the 50 ml mark with the oil to be tested and then to the 100 ml mark with solvent. The sample of oil is to be well mixed and poured into the centrifuge tube directly from a sample container previously shaken or from a circulating stream used for mixing, using precautions outlined in E.1. Stopper the tube and shake until the contents are thoroughly mixed. While filling the centrifuge tube first with the oil sample and then with the solvent gives a more accurate measurement of the oil sample volume, some crudes may cause deposits in the tip of the tube which are not easily removed. When tests indicate such a problem with a particular crude, the order of adding the crude oil and solvent to the centrifuge tube may be reversed. Immerse the tube in a bath or dry heating device and heat the contents of the tube to $49 \pm 1^\circ\text{C}$ ($120 \pm 1.8^\circ\text{F}$). Make every effort to ensure a

consistent sample temperature after whirling, shall not drop below 38°C (100°F) unless mutually acceptable comparative test has established that the results are unaffected by the lower temperatures.

2. If wax contributes to the volume of water and sediment observed, preheat the oil solvent mixture to 60°C (140°F) before first swirling; the final temperature of the mixture shall not drop below 52°C (125°F) with a water-saturated solvent of 46°C (115°F) otherwise. A heated centrifuge may be required to maintain this final temperature (see Appendix X1 for discussion on the effect of higher than recommended temperatures on the solubility of water in solvents).
3. Invert tube to ensure that the oil and the solvent are uniformly mixed. If shaking is necessary, proceed cautiously because the vapor pressure at 60°C (140°F) is approximately double than at 38°C (100°F). Place tubes in trunion cups on opposite sides of the centrifuge to establish a balanced condition, and whirl for 3 to 10 minutes depending upon the character of the sample, at a rate calculated from the equation given in C.1, sufficient to produce a relative centrifugal force (rcf) of between 500 and 800 at the tip of the whirling tubes.
4. Read and record the combined volume of water and sediment at the bottom of the tube to the nearest 0.05 ml from 0.1 to 1 ml graduation and to the nearest 0.1 above 1 ml graduation. Below 0.1 ml, estimate to the nearest 0.025 ml. If experience with the oil is limited, it is advisable to reheat the sample and return the tube to the centrifuge without agitation and repeat the operation until two consecutive readings are in agreement. Once an acceptable whirling time is established, this may be used without repeating by mutual consent.
5. With certain types of oil it is difficult to obtain a clean break between the oil layer and the separated water. In such cases one or more of the following remedies may be effective: 1) raise the temperature to 60°C (140°F); 2) shake the mixture between whirling in centrifuge just sufficiently to disperse the emulsion; 3) use a demulsifier or determine if a different type or an increased amount of demulsifier is needed; however, it should not contribute to the water and sediment; 4) use a different or increased amount of solvent and adjust reading and reporting procedure accordingly. After a satisfactory procedure for a particular type of oil has been worked out, it will ordinarily be found suitable for all samples of the same type.

G. CALCULATION

1. If a single tube is used, multiply the reading obtained according to procedure described in 6.3 by two and record the results as the percentage of water and sediment. For example, if a reading is 0.025 ml, record the percentage of water and sediment at 0.05. If a reading is 0.15 ml, record the percentage of water and sediment as 0.30. The percentages to be recorded can be read directly from a 200-part tube provided the tube contains 50 ml or 100 parts of oil. When the volume of oil is different than 50 ml or 100 parts, calculate the percent sediment and water as follows:

$$\text{Sediment and water, \%} = (S/V) \times 100$$

where:

S = volume of sediment and water, ml or parts and

V = volume of oil tested, ml or parts.

2. If two tubes are used, record the final volume of water and sediment in each tube and report

the sum of these two readings as the percentage of water and sediment. Report results lower than 0.05% either as zero or 0.05, whichever is closer. With 200-part tubes, report the average of the two percentage readings.

H. PRECISION AND ACCURACY

See Quality Assurance section.

I. REFERENCES

- 1. ASTM D96-73**
- 2. ASTM D1796-75**

DETERMINING A RESPONSE AMPLITUDE OPERATOR AND CREATING A SEA SPECTRA BOOM RESPONSE MODEL FROM TANK TEST MEASUREMENTS

In order to model a boom's motion in a harbor chop or random sea, responses to monochromatic wave forms in the test tank are mathematically combined. The first step is to measure the monochromatic responses and either (1) determine numerical values to describe the responses or (2) digitally record the response. The first option is suitable for classifying the response and report presentations. The second option is useful for constructing models.

An analytical model was developed using Lotus 1-2-3® to predict the Oil Fence response to a four component sea spectra. Four wave periods from thirteen used in tank tests were selected. They were 7, 3.6, 2.4 and 1.7 second period waves. The model was set for the user to assign wave heights to each of the four waves. The model would then calculate the contrived water surface wave form and the response of the boom apex to that water surface over a 150-second time period. The wave patterns of the water surface and boom are presented graphically as in Figure A-1. Although the water surface wave pattern portrays a sea surface wave pattern with respect to a fixed reference, the boom wave pattern is with respect to the model sea surface. The model also provides the user with maximum and minimum boom depth values. The user quickly knows if the model sea spectra has caused the boom to come out of the water or submerge at times.

The water surface wave pattern and boom response pattern are determined using the principal of superposition. The water surface wave height model is:

$$H_T = H_1 (\sin \alpha t) + H_2 (\sin \beta t) + H_3 (\sin \delta t) + H_4 (\sin \epsilon t) \quad (3)$$

Where α , β , δ , and ϵ are the encounter frequencies in radians per second (rotational frequency) of each component waves. The user specified wave heights are : H_1 , H_2 , H_3 , and H_4 . t is lapsed time in seconds. The boom depth amplitude response model is:

$$h_T = (B_1 * W_1) + (B_2 * W_2) + (B_3 * W_3) + (B_4 * W_4) \quad (4)$$

Where:

$$\begin{aligned} W_1 &= H_1 (\sin \alpha t) \\ W_2 &= H_2 (\sin \beta t) \dots \end{aligned} \quad (5)$$

and the measured boom depth amplitude response values are B_1 , B_2 , B_3 , and B_4 for the four component waves respectively.

The values of B_1 , B_2 , ... B_n combined are the response operator and are reported in table or graphical form. Values for B , the amplitude of the boom response, can be determined manually from a stripchart trace as in Figure A-2.

The easier way is to computer analyze digitally-recorded data using a Fourier Transform program and get a frequency magnitude response. The Fourier Program, Protocol Basic, transforms the time-based pressure data into frequency-based or periodicity-based data. The process begins with the output signal from pressure transmitters through data logging, file conversion, analysis and document printing.

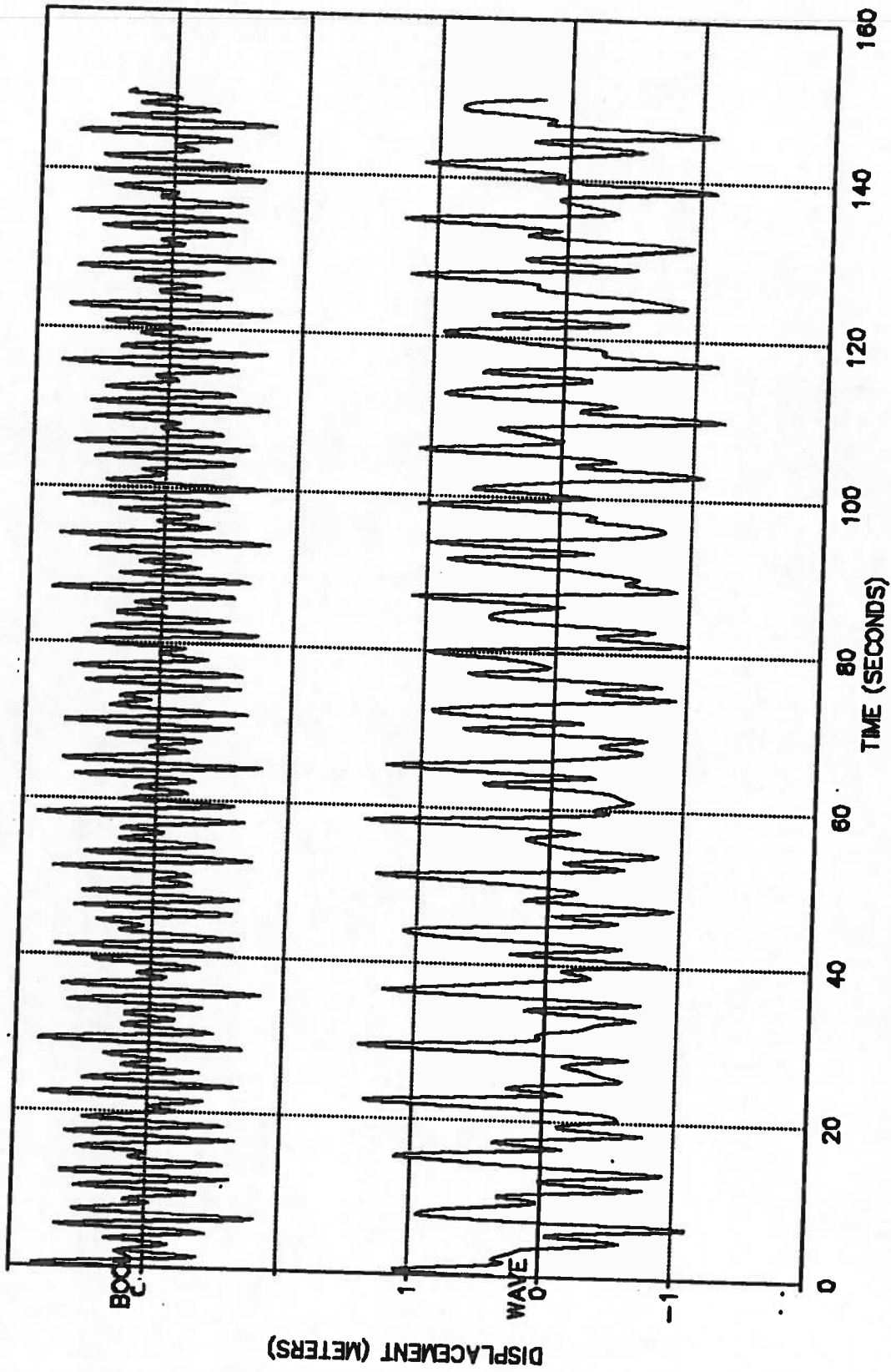


FIGURE A-1 - Boom Response Model predictions based on a four component wave spectra. The lower trace represents the surface wave the upper trace represents the boom skirt depth over the same time period.

Figures A-3, A-4 and A-5 were generated from a system calibration exercise. A pressure transmitter was attached to a shaft that oscillated up and down in a water tank. This simulated a boom skirt rising and falling in the water column. The milliamp output signal was recorded on a Rustrak Ranger II datalogger. At the completion of several more tests, the data was downloaded to a personal computer in a "PRN" format file and then "imported" into a Lotus 1-2-3® spreadsheet to generate the time-based graph. Due to the nature of the Ranger II format, the original PRN file required editing so it could be used by the Protocol Basic program. The editing was done on the spreadsheet and a second PRN file was created by "printing" a file to disk. In the case of boom motion in a monochromatic wave, the data manipulation process will result in a monochromatic response near the same frequency of the wave. The value for B is the ratio of the response magnitude to the wave magnitude at the wave frequency.

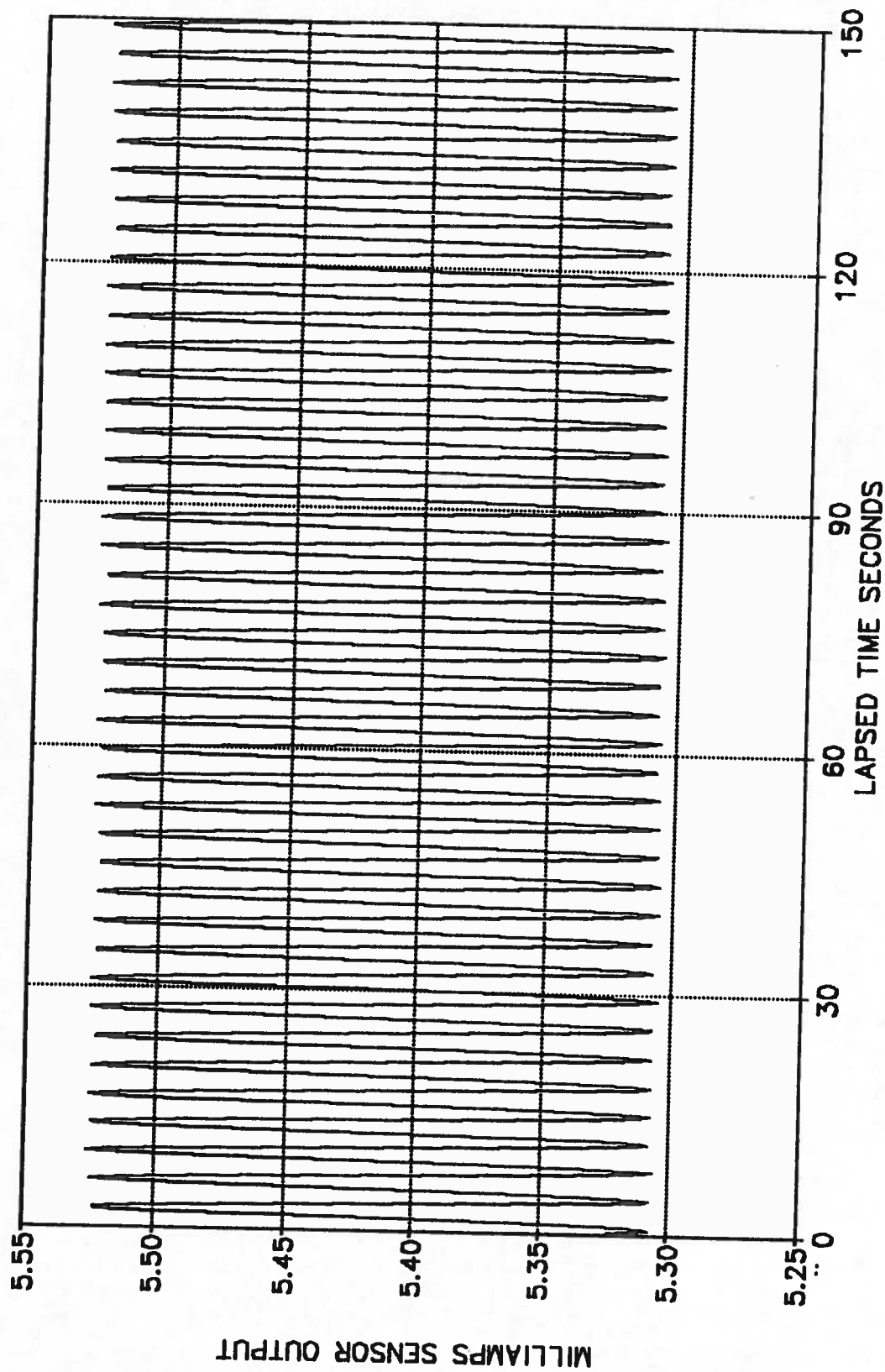


FIGURE A-3 - Time-based monochromatic signal variation from a pressure transmitter calibration exercise.

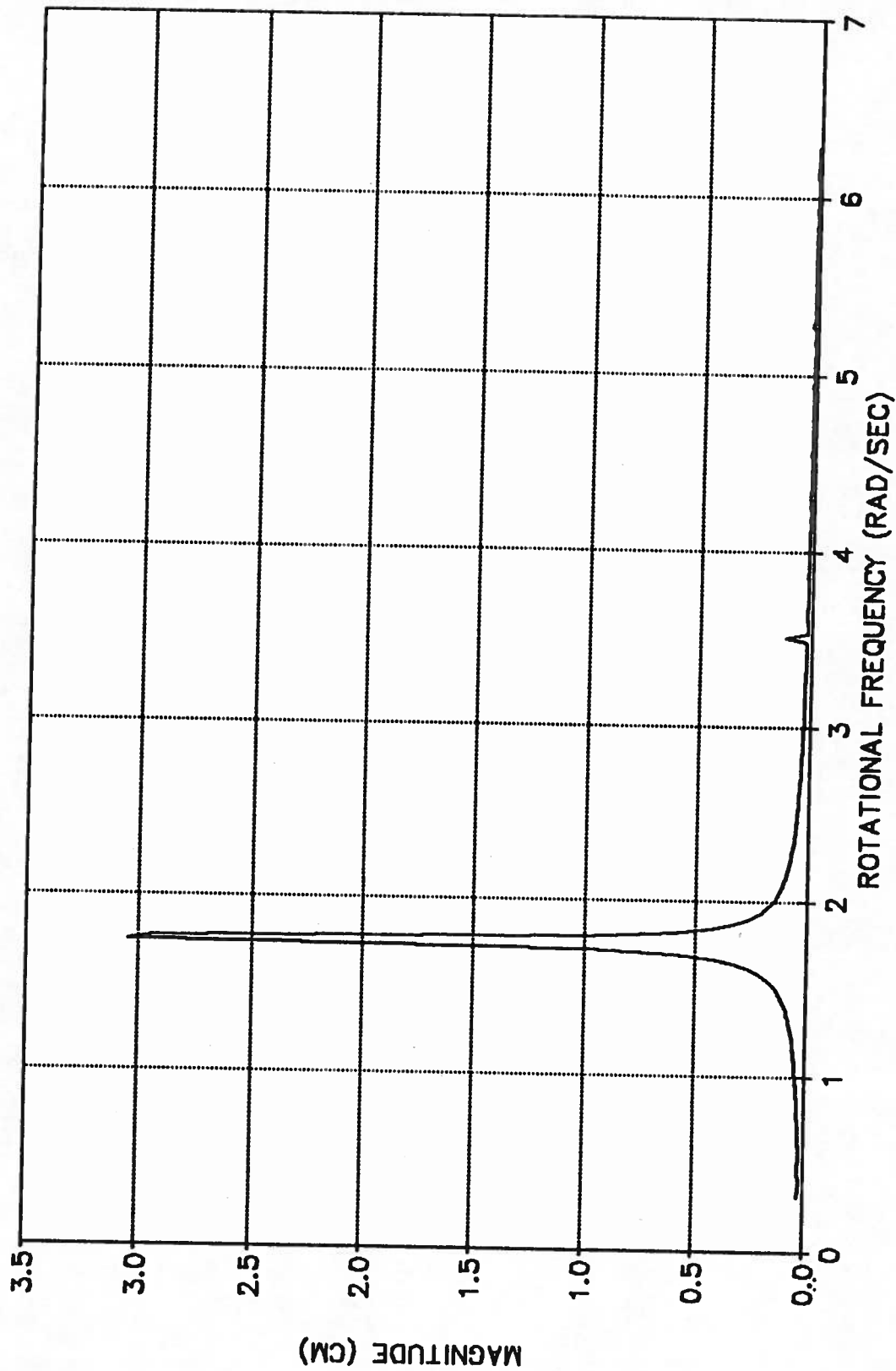


FIGURE A-4 - Frequency-based monochromatic signal graphed from Protocol Basic output.

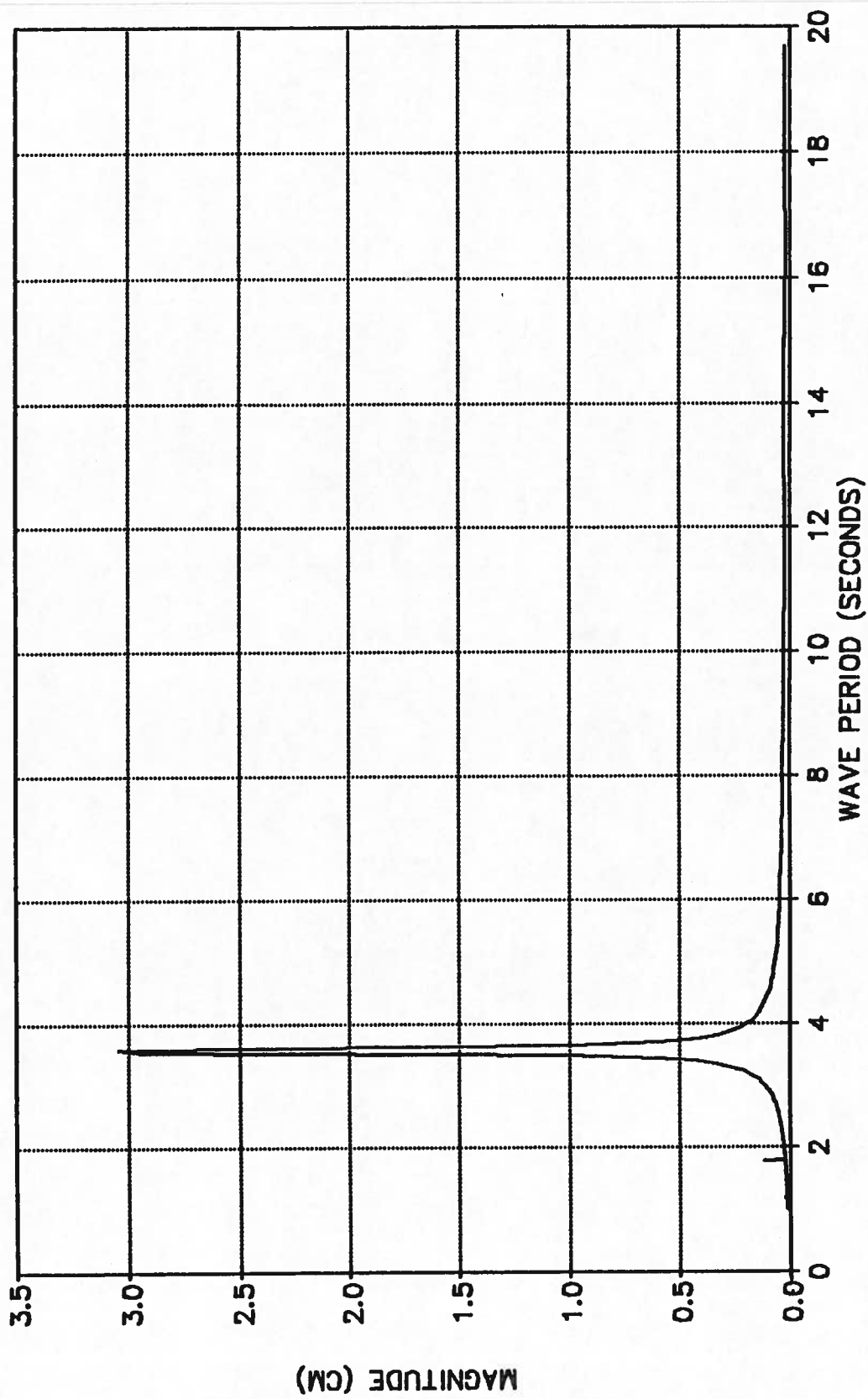


FIGURE A-5 - Periodicity-based monochromatic signal graphed from Protocol Basic output.

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10 ' PROTOCOL.BAS (PROTO4.BAS)
20 '
30 ' BASIC PROGRAM WRITTEN TO PERFORM DATA QUALIFICATION
31 ' CALCULATING IN CONJUNCTION WITH BOOM TEST PROTOCOL
32 '
33 ' THIS PROGRAM IS BELIEVED TO BE CORRECT AND FUNCTIONAL
34 ' AS WRITTEN. IT HAS NOT BEEN REVIEWED BY AN EXTERNAL
35 ' SOURCE. NO GUARENTEE OF CORRECTNESS OR COMPLETENESS
36 ' EXPLICIT, IMPLICIT OR OTHERWISE SHOULD BE ASSUMED.
50 '
60 ' WRITTEN FEBRUARY 1985 REVISED AUGUST 1987
70 '
80 ' MICHAEL BORSI
90 ' MASON & HANGER - SILAS MASON CO., INC
100 ' USEPA OHMSETT FACILITY
110 ' POST OFFICE BOX 73
120 ' LEONARDO, NJ 07737
120 ' (201) 291-0680
120 ' (201) 291-0680
130 '
131 ' REVISIONS MADE AFTER JULY 1987 ARE
132 ' MADE UNDER THE AUSPICES OF
133 ' ROY F. WESTON INC.
134 '
140 ' WRITTEN TO BE USED WITH THE IBM PERSONAL COMPUTER
150 ' USING ADVANCED BASIC (MICROSOFT VERSION 1.10).
160 ' PROGRAM REQUIRES DATA TO BE STORED IN DISC FILE
170 ' IN DIMENSIONAL FORMAT. PROGRAM REQUIRES APPROXIMATELY 60K BYTES RAM.
180 ' FILE MUST CONTAIN AT LEAST 1024 DATA POINTS LOGGED AT EQUAL TIME
190 ' TIME INTERVALS FOR COMPLETE ANALYSIS, DATA MUST BE GREATER"
200 ' GREATER THAN -9999 AND LESS THAN 9999. DATA NEED NOT BE INTIGER
210 '
220 ' DATA FILE IS SUBJECTED TO ALL REDUCTIONS OUTLINED IN
230 ' -----PROPOSED-----
240 ' TEST PROCEDURES FOR THE EVALUATION OF OFFSHORE OIL-SPILL
250 ' CONTROL BOOMS. MB. VERSION O. DATED 16 SEPTEMBER 1985. 40 PAGES.
260 ' PROGRAM IS APPLICABLE TO SECTIONS 3.4.4.2 THROUGH 3.4.4.3.2 INCLUSIVE
270 '
280 ' PROGRAM AND STANDARD WRITTEN UNDER THE AUSPICES OF THE
290 ' OHMSETT INTERAGENCY TECHNICAL COMMITTEE. THE COMMITTEE IS COMPRISED
300 ' OF THE US MINERALS MANAGEMENT SERVICE, THE US COAST GUARD, THE US EPA,
310 ' THE US NAVY, AND THE CANADIAN ENVIRONMENTAL PROTECTION SERVICE.
320 '
330 ' BASED ON INFORMATION IN:
340 '
350 ' BENDAT, J.S. AND PIERSON R.G., RANDOM DATA: ANALYSIS AND
360 ' MEASUREMENT PROCEDURES, WILEY-INTERSCIENCE NEW YORK, NY.,
370 ' 1971 407 PAGES.
380 '
390 ' COMSTOCK, JOHN P. (PRINCIPLE EDITOR), PRINCIPLES OF NAVAL
400 ' ARCHITECTURE, SOC. OF NAVAL ARCHITECTS AND MARINE ENGINEERS,
410 ' NEW YORK, NY., 1980 827 PAGES.
420 '
430 ' MASON, R.D. et. al., STATISTICS an Introduction, BRACE
440 ' MARCOURT JOVANOVIH INC., NEW YORK, NY., 626 PAGES.
450 '
460 ' BAUMEISTER AND MARKS, STANDARD HANDBOOK FOR MECHANICAL ENGINEERS,
470 ' 7th EDITION, MCGRAW HILL, NEW YORK, NY., 1967
480 '
490 ' STANLEY, W.D., AND PETERSON, S.J. "FAST FOURIER TRANSFORMS ON
500 ' YOUR HOME COMPUTER", BYTE MAGAZINE VOL 3 NUMBER 12 DEC. 1978
510 ' PP 14-25.
520 '
530 ' COLOR 15,1,1 :CLS : KEY OFF : OPTION BASE 1 : WIDTH "LPT1:",130
540 '
550 ' DIMENSION ARRAYS
560 '

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1170 IF DUMMY<>0 THEN 1210
1180 PRINT "DEFAULT DRIVE ASSUMED ";
1190 INPUT "IS THIS CORRECT (Y/N) ";ANSWER$
1200 ANSWER$=LEFT$(ANSWER$,1) : IF ANSWER$<>"Y" THEN 1140
1210 DUMMY=INSTR(PWRFIL$,".") : IF DUMMY=LEN(PWRFIL$)-3 AND DUMMY<>0 THEN 125
0
1220 PRINT "EXTENSION MISSING OR LESS THAN 3 CHAR'S "
1230 INPUT "CORRECT AS IS (Y/N) ";ANSWER$ : ANSWER$=LEFT$(ANSWER$,1)
1240 IF ANSWER$<>"Y" THEN 1140
1250 ON ERROR GOTO 4720
1260 INPUT "DO YOU WISH TO SAVE HISTOGRAM DATA TO THE DISK ";HANS$
1261 HANS$=LEFT$(HANS$,1)
1262 IF HANS$="N" THEN 1269 ELSE IF HANS$<>"Y" THEN PRINT "Y/N ONLY" : GOTO 1
260 ELSE INPUT "FILE NAME ";HFIL$
1263 DUMMY=INSTR(HFIL$,".")
1264 IF LEN(HFIL$)-DUMMY=3 THEN 1267
1265 INPUT "EXTENSION IS MISSING OR LESS THAN 3 CHAR'S. OK?";HANS$
1266 HANS$=LEFT$(HANS$,1) : IF HANS$="N" THEN HANS$="" : GOTO 1262
1267 DUMMY=INSTR(HFIL$,".")
1268 IF DUMMY=0 THEN PRINT "DEFAULT DRIVES ASSUMED"
1269 OPEN FILE0$ FOR INPUT AS #1
1270 IF HMT$<>"Y" THEN 1279
1271 FOR I=1 TO 19
1272 LINE INPUT #1,A$ : PRINT A$
1273 NEXT I
1279 IF EOF(1) THEN 1310
1280 IF HMT$<>"Y" THEN INPUT #1,DUMMY ELSE INPUT #1,DUMMY,DUMMY
1290 NPOINT=NPOINT+1
1300 GOTO 1279
1310 CLOSE
1320 PWR2=LOG(NPOINT)/LOG(2) : IPWR2=INT(PWR2)
1321 IF IPWR2>10 THEN IPWR2=10
1322 NPOINTS=INT(2^IPWR2)
1330 INPUT "WHAT IS THE TIME SPACING (SEC) ";DT
1340 INPUT "WHAT IS THE UNIT OF THE DATA SET ";UNIT$
1350 IF UNIT$="FEET" OR UNIT$="FOOT" OR UNIT$="FT" THEN G=32.174
1360 IF UNIT$="M" OR UNIT$="METER" THEN G=9.810001
1370 IF UNIT$="CM" OR UNIT$="CENTIMETER" THEN G=981
1380 IF UNIT$="BCD" THEN G=1
1390 IF G<>0 THEN 1420
1400 PRINT "WITH UNITS OF ";UNIT$," I DO NOT KNOW THE VALUE OF G"
1410 INPUT "WHAT IS THE VALUE OF G (1 FOR NOT IMPORTANT)";G
1420 TOTTIM=NPOINTS*DT : RES=1/NPOINTS/DT
1430 IF NPOINT=NPOINTS THEN 1650
1440 PRINT "THIS PROGRAM WILL ANALYZE ONLY AN EXACT POWER OF 2 DATA POINTS"
1441 PRINT " TO A MAXIMUM OF 1024 DATA POINTS"
1450 PRINT "THE SPECIFIED FILE (";FILE0$;) CONTAINS ";NPOINT
1460 PRINT "THE MAXIMUM POWER OF 2 IS ";INT(PWR2);" OR ";NPOINTS
1480 PRINT "PROGRAM REQUIRES APPROXIMATELY 8:30 FROM THIS POINT"
1490 IF NPOINT=NPOINTS THEN NSKIP=0 : GOTO 1650
1500 PRINT "THE PROGRAM REQUIRES EXACTLY ";NPOINT;" DATA POINTS "
1510 PRINT "THE SPECIFIED FILE CONTAINS ";NPOINT
1520 PRINT "YOU CAN (1) SELECT DATA STARTING WITH #1"
1530 PRINT " (2) SELECT DATA ENDING WITH #";NPOINT
1540 PRINT " (3) CHOOSE THE MIDDLE MOST POINTS "
1541 PRINT " (4) SPECIFY THE NUMBER OF DATA POINT TO SKIP AT THE START
OF THE FILE"
1550 PRINT " (5) TERMINATE THE PROGRAM HERE ";
1560 INPUT "SELECTION : ";ANSWER
1570 IF ANSWER <> INT (ANSWER) OR ANSWER<1 OR ANSWER>5 THEN CLS : GOTO 1500
1580 IF ANSWER=1 THEN NSKIP=0 : GOTO 1650
1590 IF ANSWER=2 THEN NSKIP=NPOINT-NPOINTS-1 : GOTO 1650
1600 IF ANSWER=3 THEN NSKIP=(NPOINT-NPOINTS)\2 : GOTO 1650
1601 IF ANSWER=5 THEN PRINT "TERMINATED AT USERS REQUEST ":END
1610 IF ANSWER=4 THEN INPUT "SKIP HOW MANY POINTS ";NSKIP
1620 IF NPOINT-NPOINTS->NSKIP THEN 1650

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1630 BEEP : PRINT "IMPOSSIBLE REQUEST. MAXIMUM SKIP IS ",NPOINT-NPOINTS : GO
TO 1610
1650 OPEN FILEO$ FOR INPUT AS #1
1651 BEGIN$=DATE$+" "+TIME$
1652 IF HMT$<>"Y" THEN 1660
1653 FOR I=1 TO 19 : LINE INPUT #1,DUMMH$: NEXT I 'SKIP HERMIT HEADER
1660 IF NSKIP=0 THEN 1700
1670 FOR I=1 TO NSKIP
1680 IF HMT$<>"Y" THEN INPUT #1,DUMMY ELSE INPUT #1,DUMMY,DUMMY

1690 NEXT I
1700 FOR I=1 TO NPOINTS 'SKIP DATA AS SPECIFIED
1710 IF EOF(1) THEN 1750 'INPUT DATA FROM FILE
1730 IF HMT$<>"Y" THEN INPUT #1,DAT(I) ELSE INPUT #1,DUMMY,DAT(I)
1740 NEXT I
1750 CLOSE
1760 IF ANS$<>"Y" THEN 1810
1770 FOR I=1 TO NPOINTS
1780 DAT(I)=DAT(I)*CSLOPE+CINTER 'USER-DEFINED CONVERSION
1790 NEXT I
1810 FOR I=1 TO NPOINTS
1820 SUMDAT=SUMDAT+DAT(I) 'SUM OF DATA
1830 SUM2DAT=SUM2DAT+DAT(I)^2 'SUM OF SQUARES OF DATA
1840 SUMTIM=(I-1)*DT+SUMTIM 'SUM OF ELAPSED TIMES
1850 SUM2TIM=SUM2TIM+((I-1)*DT)^2 'SUM OF SQUARES OF TIMES
1860 MIXSUM=MIXSUM+DAT(I)*(I-1)*DT 'MIXED SUM
1870 IF MINDAT>DAT(I) THEN MINDAT=DAT(I) 'DATA MINIMUM
1880 IF MAXDAT<DAT(I) THEN MAXDAT=DAT(I) 'DATA MAXIMUM
1890 NEXT I
1900 AVEDAT=SUMDAT/NPOINTS 'DATA AVERAGE
1910 VARDAT=(SUM2DAT-SUMDAT^2/NPOINTS)/(NPOINTS-1) 'DATA VARIANCE
1920 STDDAT=SQR(ABS(VARDAT)) 'DATA STANDARD DEVIATION
1930 RANDAT=MAXDAT-MINDAT 'DATA RANGE
1940 RMSDAT=SQR(SUM2DAT/NPOINTS) 'ROOT MEAN SQUARE
1950 MSQDAT=SUM2DAT/NPOINTS 'MEAN SQUARE
1960 SLOPE=(NPOINTS*MIXSUM-SUMTIM*SUMDAT)/(NPOINTS*SUM2TIM-SUMTIM^2)
1970 INTER=(SUMDAT-SLOPE*SUMTIM)/NPOINTS
1980 DUMMY=(NPOINTS*SUM2TIM-SUMTIM^2)*(NPOINTS*SUM2DAT-SUMDAT^2)
1990 COCOR=(NPOINTS*MIXSUM-SUMDAT*SUMTIM)/SQR(ABS(DUMMY))
2000 TSTAT=SQR(NPOINTS-2)*COCOR/SQR(ABS(1-COCOR^2))
2001 OPEN OUT1$ FOR APPEND AS #1
2010 PRINT #1, CHR$(12),CHR$(15); 'NEW PAGE, COND PRINT
2011 PRINT #1, " PRELIMINARY ANALYSIS PER OHMSETT TECHNIQUES" : PRINT #1,
2020 PRINT #1, " PRELIMINARY STATISTICS ",DETREND2$
2030 PRINT #1, " FILE NAME "
2040 PRINT #1, USING " POINTS AVAILABLE " " ,FILEO$
2050 PRINT #1, USING " POINTS ANALYZED " " ,NPOINT
2060 PRINT #1, USING " LEADING POINTS SKIPPED " " ,NPOINTS
2061 PRINT #1, USING " TRAILING POINTS SKIPPED " " ,NSKIP
NSKIP PRINT #1, USING " " " ,NPOINT-NPOINTS-
2080 PRINT #1, USING " DATA AVERAGE " " ,AVEDAT
2090 PRINT #1, USING " DATA MAXIMUM " " ,MAXDAT
2095 PRINT #1, USING " DATA MINIMUM " " ,MINDAT
2100 PRINT #1, USING " DATA RANGE " " ,RANDAT
2120 PRINT #1, USING " DATA STANDARD DEVIATION " " ,STDDAT
2130 PRINT #1, USING " DATA VARIANCE " " ,VARDAT
2140 PRINT #1, USING " DATA MEAN SQUARE " " ,MSQDAT
2150 PRINT #1, USING " DATA ROOT MEAN SQUARE " " ,RMSDAT
2151 PRINT #1, USING " DATA ANALYSIS BEGAN "
",BEGIN$
2160 PRINT #1, SPACE$(5);,FOR I=1 TO 65 : PRINT #1, " ";NEXT I : PRINT #1,
2170 PRINT #1, SPACE$(5);,FOR I=1 TO 65 : PRINT #1, " ";NEXT I : PRINT #1,
PRINT #1,
2180 PRINT #1, " LEAST-SQUARES REGRESSION RESULTS (TIME ON DATA)"
2190 PRINT #1, SPACE$(5);,FOR I=1 TO 33 : PRINT #1, " ";NEXT I : PRINT #1,
2200 PRINT #1, USING " SLOPE " " ,SLOPE,

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2210 PRINT #1, " ",UNIT$, " /SEC"
2220 PRINT #1, USING " INTERCEPT ###.###",INTER,
2230 PRINT #1, " ",UNIT$
2240 PRINT #1, USING " COEFFICIENT OF CORRALATION ##.###",COCOR
2250 PRINT #1, USING " STUDENT T-TEST STATISTIC ###.###",TSTAT
2260 PRINT #1, : PRINT #1, " LEAST-SQUARES REGRESSION SHOWS THAT LINEAR
TREND ",
2270 IF ABS(TSTAT)>1.96 THEN PRINT #1, "IS "; GOTO 2300
2280 IF ABS(TSTAT)<1 THEN PRINT #1, "IS NOT "; GOTO 2300 ELSE PRINT "MAY BE
",
2300 PRINT #1, "SIGNIFICANT"
2310 IF FORCE$="Y" THEN PRINT #1, " FORCED"; GOTO 2320 ELSE IF DETREND1$
="N" OR ABS(TSTAT)<1 THEN 2400
2320 PRINT #1, " AUTOMATID DETREND IS ON --- RECOMPUTING" : FORCE$="Y"
2321 CLOSE 1
2330 FOR I=1 TO NPOINTS
2340 DAT(I)=DAT(I)-(INTER+I*DT*SLOPE) 'REMOVE TIME TREND
2350 NEXT I
2360 SUMDAT=0 : SUM2DAT=0 : SUMTIM=0 : SUM2TIM=0 : MAXDAT=-9999
2370 HINDAT=9999 : MIXSUM=0 : DETREND1$="" : DETREND2$="DE-TRENDED DATA"
2380 GOTO 1810
2400 :
2410 : ANALYSIS OF VARIANCE
2420 :
2430 FOR J=1 TO 16
2440 FOR I=1 TO INT(NPOINTS/16)
2450 ANO(J,I)=DAT((J-1)*INT(NPOINTS/16)+I)
2460 NEXT I
2470 NEXT J
2480 FOR J=1 TO 16
2490 COLTOT(J)=0
2500 COLSTD(J)=0
2510 FOR I=1 TO INT(NPOINTS/16)
2520 COLTOT(J)=COLTOT(J)+ANO(J,I)
2530 COLSTD(J)=COLSTD(J)+ANO(J,I)^2
2540 NEXT I
2550 NEXT J
2560 FOR I=1 TO 16
2570 SSQTRT=SSQTRT+COLTOT(I)^2/INT(NPOINTS/16)
2580 COLSTD(I)=SQR(ABS((COLSTD(I)-COLTOT(I)^2/INT(NPOINTS/16))/INT(NPOINT
S/16)-1))
2590 NEXT I
2600 SSQERR=SSQTRT
2610 SSQTRT=SSQTRT-(SUMDAT)^2/NPOINTS
2620 SSQERR=SUM2DAT-SSQERR
2630 ANOVAF=(SSQTRT/15)/(SSQERR/(NPOINTS-16))
2640 SSQTOT=SUM2DAT-(SUMDAT^2/NPOINTS)
2650 PRINT #1, SPACE$(5);:FOR I=1 TO 65 : PRINT #1, " ";:NEXT I : PRINT #1,
2660 PRINT #1, SPACE$(5);:FOR I=1 TO 65 : PRINT #1, " ";:NEXT I : PRINT #1,
2670 PRINT #1, " ANOVA TEST RESULTS ";DETREND2$
2680 PRINT #1, " SOURCE SS df MS
F"
2690 PRINT #1, SPACE$(5);:FOR I=1 TO 65 : PRINT #1, " ";:NEXT I : PRINT #1,
2700 PRINT #1, USING FMT1$," TOTAL ",SSQTOT,NPOINTS-1,SSQTOT/(NPOINTS
-1)
2710 PRINT #1, USING FMT4$," TIME ",SSQTRT,15,SSQTRT/15,ANOVAF
2720 PRINT #1, USING FMT1$," RESIDUAL ",SSQERR,(NPOINTS-16-1),SSQTOT/(NP
OINTS-16-1)
2730 PRINT #1, :PRINT #1, SPACE$(5);:FOR I=1 TO 65 : PRINT #1, " ";:NEXT I :
PRINT #1, : PRINT #1,
2740 PRINT #1, " ANALYSIS OF VARIANCE INDICATES THAT THE DATA SETS ARE ";
2750 IF ANOVAF>1.67 THEN PRINT #1, "NOT ";
2760 PRINT #1, "EQUIVELANT"
2761 PRINT #1, " THERE WERE ",16*INT(NPOINTS/16)," POINTS USED IN THE ANO
VA TEST"
2762 PRINT #1, " THERE WERE ",INT(NPOINTS/16)," POINTS IN EACH OF THE 16

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```

SUBSETS"
2770 PRINT #1, SPACE$(5);;FOR I=1 TO 65 : PRINT #1, "_";NEXT I:PRINT #1,
2780 PRINT #1, "          SET STATISTICS "
2790 PRINT #1, SPACE$(5);;FOR I=1 TO 14 : PRINT #1, "_";NEXT I : PRINT #1,
2800 PRINT #1, USING"\          \          \          \" SET","AVERAGE"
,"STD.DEV"
2810 FOR I=1 TO 16
2820 PRINT #1, USING "      ##      ###.###      ###.###"; I,COLTOT(I)/INT
(NPOINTS/16),COLSTD(I)
2830 NEXT I
2840 '
2850 ' NORMALITY TEST
2860 '
2870 DATA -100,-1.862,-1.534,-1.318,-1.151,-1.01,-.887
2880 DATA -.776,-.675,-.579,-.489,-.402,-.319,-.237,-.157
2890 DATA -.078,0,.078,.157,.237,.319,.402,.489,.579,.675
2900 DATA .776,.887,1.01,1.151,1.318,1.534,1.862
2910 FOR I=1 TO 32
2920 READ HALPHA(I)                                'CUMULATIVE NORMAL VALUES
2930 HUPPER(I)=AVEDAT-HALPHA(I)*STDDAT              'LOWER LIMITS OF RANGES
2940 NEXT I
2950 FOR I=2 TO 32
2960 HLOWER(I-1)=HUPPER(I)                          'UPPER LIMITS OF RANGES
2970 NEXT I
2980 HLOWER(32)=AVEDAT-100*STDDAT                  'EXTREME LOWER LIMIT
2990 FOR J=1 TO NPOINTS
3000 FOR I=1 TO 32
3010 IF DAT(J)<=HLOWER(I) THEN 3050                'GROUP DATA
3020 IF DAT(J)>HUPPER(I) THEN 3050
3030 HCOUNT(I)=HCOUNT(I)+1
3040 GOTO 3060
3050 NEXT I
3060 NEXT J
3070 PRINT #1, CHR$(12);"      NORMALITY RESULTS ";DETREND2$
3080 PRINT #1, SPACE$(5);;FOR I=1 TO 18 : PRINT #1, "_";NEXT I : PRINT #1,
3090 PRINT #1, "      INTERVAL","      ALPHA","      LOWER","      UPPER","      C
OUNT","CHI"
3100 PRINT #1, "      NUMBER","","      LIMIT","      LIMIT","","SQUARE"
3110 PRINT #1, SPACE$(5);;FOR I=1 TO 75 : PRINT #1, "_";NEXT I:PRINT #1,
3120 DUMMY=0
3130 FOR I=1 TO 32
3140 DUMMY=DUMMY+HCOUNT(I)
3150 PRINT #1, USING FMT2$,I,HALPHA(33-I),HLOWER(I),HUPPER(I),HCOUNT(I),
(HCOUNT(I)-(NPOINTS/32))^2/(NPOINTS/32);
3160 IF DUMMY>NPOINTS/2 THEN PRINT #1, MED$;: MED$=""
3170 IF HLOWER(I)<AVEDAT AND HUPPER(I)=>AVEDAT THEN PRINT #1, " -AVERAGE"
/
3180 PRINT #1,
3190 IF HCOUNT(I)<5 THEN WARNING$="ONE OR MORE RANGES HAVE LESS THAN FIV
E --- CHI-SQUARE TEST MAY BE MISAPPLIED
3200 NEXT I
3210 PRINT #1, SPACE$(5);;FOR I=1 TO 75 : PRINT #1, "_";NEXT I : PRINT #1,
3211 HEXPECT=NPOINTS/32                          'EXPECTED POINTS PER DIVIS
ION
3220 FOR I=1 TO 32
3230 PCOUNT=PCOUNT+HCOUNT(I)
3240 CHISQR=CHISQR+(HCOUNT(I)-HEXPECT)^2/HEXPECT
3250 NEXT I
3260 PRINT #1, "      CHI SQUARE IS ";CHISQR;" AND ";
3270 PRINT #1, "TOTAL POINTS ACCOUNTED FOR ";PCOUNT
3280 PRINT #1, "      DATA ";
3290 IF CHISQR>26 THEN PRINT #1, "FAILS "; ELSE PRINT #1,"PASSES ";
3300 PRINT #1," CHI-SQUARE TEST FOR NORMALITY " :PCOUNT=0
3301 PRINT #1,"      EXPECTED NUMBER OF POINTS PER DIVISION IS ";HEXPECT
3310 PRINT #1, SPACE$(5);WARNING$ : MED$="-MEDIAN"
3320 PRINT #1, SPACE$(5);;FOR I=1 TO 75 : PRINT #1, "_";NEXT I : PRINT #1,

```

```

3330 IF CHISQR<26 THEN 3560
3340 FOR I=1 TO 32
3350 HCOUNT(I)=0 : HLOWER(I)=(I-1)*RANDAT/32+MINDAT
3360 HUPPER(I)=I*RANDAT/32+MINDAT
3370 NEXT I
3380 FOR I=1 TO NPOINTS
3390 DUMMY=INT((DAT(I)-MINDAT)*32/RANDAT)+1
3400 IF DUMMY>32 THEN DUMMY=32
3410 HCOUNT(DUMMY)=HCOUNT(DUMMY)+1
3420 NEXT I
3430 PRINT #1, CHR$(12);" HISTOGRAM DATA ";DETREND2$
3440 PRINT #1, SPACE$(5);:FOR I=1 TO 14 : PRINT #1, "_";:NEXT I : PRINT #1,
3450 PRINT #1, " INTERVAL", " LOWER", " UPPER", " COUNT"
3460 PRINT #1, " NUMBER", " LIMIT", " LIMIT"
3470 PRINT #1, SPACE$(5);:FOR I=1 TO 55 : PRINT #1, "_";:NEXT I : PRINT #1,
3471 IF HFIL$<>" THEN OPEN HFIL$ FOR APPEND AS #2
3480 FOR I=1 TO 32
3490 PRINT #1, USING FMT3$,I,HLOWER(I),HUPPER(I),HCOUNT(I);
3491 IF HFIL$<>" THEN PRINT #2, USING FMT3$,I,HLOWER(I),HUPPER(I),HCOUNT
3500 PCOUNT=PCOUNT+HCOUNT(I)
3510 IF PCOUNT>NPOINTS/2 THEN PRINT #1, MED$;:MED$=""
3520 IF HLOWER(I)<AVEDAT AND HUPPER(I)=>AVEDAT THEN PRINT #1, " -AVERAGE"
3530 PRINT #1,
3540 NEXT I
3541 IF HFIL$<>" THEN CLOSE 2
3550 PRINT #1, SPACE$(5);:FOR I=1 TO 55 : PRINT #1, "_";:NEXT I : PRINT #1,
3560
3570 RUN TEST
3580
3590 FOR I=1 TO 100
3600 FOR J=1 TO INT(NPOINTS/100)
3610 RCOUNT(I)=RCOUNT(I)+DAT((I-1)*INT(NPOINTS/100)+J)^2
3620 NEXT J
3630 RCOUNT(I)=RCOUNT(I)/INT(NPOINTS/100)
3640 NEXT I
3650 FOR I=1 TO 100
3660 RCOUNT(I)=SGN(RCOUNT(I)-MSQDAT)
3670 NEXT I
3680 FOR I=2 TO 100
3690 IF RCOUNT(I)=RCOUNT(I-1) OR RCOUNT(I)=0 THEN 3710
3700 IF RCOUNT(I)>0 THEN PRUNS=PRUNS+1 ELSE NRUNS=NRUNS+1
3710 NEXT I
3720 TOTRUN=NRUNS+PRUNS
3730 PRINT #1, : PRINT #1, " STATIONARITY TEST RESULTS ";DETREND2$
3740 PRINT #1, SPACE$(5);:FOR I=1 TO 25 : PRINT #1, "_";:NEXT I : PRINT #1,
3750 PRINT #1, " RUN TEST SHOWS ";TOTRUN;"RUNS"
3760 PRINT #1, " THERE WERE ";PRUNS;"POSITIVE RUNS AND ";
3770 PRINT #1, "AND ";NRUNS;" NEGATIVE RUNS"
3780 PRINT #1, " STATIONARITY SHOULD ";
3790 IF TOTRUN>61 OR TOTRUN<40 THEN PRINT #1, "NOT ";
3800 PRINT #1, "BE PRESUMED IN THE DATA SET"
3810 PRINT #1, " STATIONARITY TESTED USING MEAN SQUARE OF ";INT(NPOINTS/1
00);" POINTS"
3830 PRINT #1, CHR$(12);" PERIODICITY TEST RESULTS"
3840 PRINT #1, SPACE$(5);:FOR I=1 TO 23 : PRINT #1, "_";:NEXT I : PRINT #1,
3850
3860 FOURIER TRANSFORMATION
3870
3880 FOR I=1 TO NPOINTS
3890 DAT(I)=(DAT(I)-AVEDAT)/NPOINTS 'FFT OF DIFFERENCES FROM
3900 IMG(I)=0 'FROM AVERAGE SO THAT OC
3910 NEXT I 'COMPONENT EQUALS ZERO
3920 I1=NPOINTS/2 : I2=1 : NU=2*PI/NPOINTS
3930 FOR I=1 TO IPWR2

```



```

3940      I3=0 : I4=I1
3950      FOR K=1 TO I2
3960          X=I3/I1
3970          GOSUB 4540
3980          I5=Y
3990          Z1=COS(I5*NU) : Z2=-SIN(I5*NU)
4000          .FOR M=I3+1 TO I4
4010              A1=DAT(M) : A2=IMG(M)
4020              B1=Z1*DAT(M+I1)-Z2*IMG(M+I1)
4030              B2=Z2*DAT(M+I1)+Z1*IMG(M+I1)
4040              DAT(M)=A1+B1 : IMG(M)=A2+B2
4050              DAT(M+I1)=A1-B1 : IMG(M+I1)=A2-B2
4060          NEXT M
4070          I3=I3+2*I1 : I4=I4+2*I1
4080      NEXT K
4090      I1=I1/2 : I2=I2*2
4100      NEXT I
4101      CLOSE 1
4110      IF MAGFIL$<>"NONE" THEN OPEN MAGFIL$ FOR APPEND AS #1
4120      IF PWRFIL$<>"NONE" THEN OPEN PWRFIL$ FOR APPEND AS #2
4130      FOR I=1 TO NPOINTS/2+1
4140          X=I-1 : GOSUB 4630
4150          MAG(I)=2*DUMMY
4160          IF MAG(I)>MAXMAG THEN MAXMAG=MAG(I) : MXMAGF=I*RES
4170          PWR(I)=2*(DUMMY)^2/NPOINTS*DT
4180          IF PWR(I)>MAXPWR THEN MAXPWR=PWR(I) : MXPWRF=I*RES
4190          IF MAGFIL$<>"NONE" THEN PRINT #1,I-1,MAG(I)
4200          IF PWRFIL$<>"NONE" THEN PRINT #2,I-1,PWR(I)
4210      NEXT I
4220      CLOSE
4221      OPEN OUT1$ FOR APPEND AS #1
4230      SIMP(1)=1 : SIMP((NPOINTS/2)+1)=1
4240      FOR I=2 TO NPOINTS/2 STEP 2
4250          SIMP(I)=4 : SIMP(I+1)=2
4260      NEXT I
4270      FOR I=1 TO NPOINTS/2+1
4280          HAGARO=HAGARO+SIMP(I)*MAG(I)^2/6
4290          HAGAR2=HAGAR2+SIMP(I)*MAG(I)^2/6*((1-.5)*RES*2*PI)^2
4300          HAGAR4=HAGAR4+SIMP(I)*MAG(I)^2/6*((1-.5)*RES*2*PI)^4
4310      NEXT I
4320      PRINT #1, "      MAGNITUDE SPECTRUM FILE : ",MAGFIL$
4330      PRINT #1, "      POWER DENSITY SPECTRUM FILE : ",PWRFIL$
4340      PRINT #1, "      ",NPOINTS," DATA POINTS ANALYZED WITH ",DT,"SECOND SPACI
NG"
4350      PRINT #1, "      RESULTING INTERNAL SPECTRAL RESOLUTION ",RES," Hz"
4360      PRINT #1, "      RESULTING NYQUIST FREQUENCY ",(NPOINTS/2-1)*RES," Hz"
4370      PRINT #1, "      OTHER HARMONICS WITH POWER MAGNITUDES > MAX/3"
4380      PRINT #1, "      HARMONIC","FREQUENCY","MAGNITUDE"
4390      FOR I=1 TO NPOINTS/2
4400          IF PWR(I)<MAXPWR/3 AND PWR(I)<>MAXPWR THEN 4420
4410          PRINT #1, "      ",I-1,(I-1)*RES,PWR(I)
4420      NEXT I
4430      PRINT #1, "      MAXIMUM MAGNITUDE LOCATED AT ",MXMAGF," Hz"
4440      PRINT #1, "      MAXIMUM POWER DENSITY LOCATED AT ",MXPWRF," Hz " ,MAXPW
R," ",UNIT$,"^2-SEC"
4450      PRINT #1, USING "      ZERO-TH MOMENT = ####.###",HAGARO
4460      PRINT #1, USING "      SECOND MOMENT = ####.###",HAGAR2
4470      PRINT #1, USING "      FOURTH MOMENT = ####.###",HAGAR4 : PRINT #1,
4480      PRINT #1, : PRINT #1, "      4.0*",CHR$(251),"E =",41*SQR(HAGARO),UNIT$
4490      PRINT #1, "      2",CHR$(227),CHR$(251),"(E/M2) = ",2*PI*SQR(HAGARO/HAGAR
2)," SEC
4500      PRINT #1, "      2",CHR$(227),"g",CHR$(251),"(E/M4) = ",2*PI*G*SQR(HAGARO/H
AGAR4),UNIT$
4510      PRINT #1, "      ***** g= ",G, " ",UNIT$"/SEC^2"
4520      PRINT #1, "      DATA ANALYSIS COMPLETE AT ",DATE$," ",TIME$
4521      CLOSE #1

```

```

4530     END
4540 '
4550 'SCRAMBLER SUBROUTINE
4560 '
4570     Y=0 : N1=NPOINTS
4580     FOR W=1 TO IPWR2
4590         N1=N1/2
4600         IF X>N1 THEN Y=Y+2*(W-1) : X=X-N1
4610     NEXT W
4620     RETURN
4630 '
4640 ' MAGNITUDE SUBROUTINE
4650 '
4660     GOSUB 4560
4670     DUMMY=SQR(DAT(Y+1)^2+IMG(Y+1)^2)
4680     RETURN
4690 '
4700 ' ERROR TRAPPING SUBROUTINES
4710 '
4720     BEEP : CLS : BEEP : BEEP
4730     IF ERR=76 THEN PRINT DRIVE0$;" IS NOT VALID"
4740     IF ERR<>61 THEN 4770
4750     PRINT "DISK FULL -- OUTPUT FILE NAMES CHANGED TO NONE"
4760     MAGFIL$="NONE" : PWRFIL$="NONE" : CLOSE: RESUME
4770     IF ERR<>71 THEN 4790
4780     PRINT "DISK DRIVE NOT READY -- FILE OUTPUT DELETED" : GOTO 4760
4790     IF ERR<>70 THEN 4820
4800     PRINT "DIST IS WRITE PROTECTED -- FILE OUTPUT DELETED " : GOTO 4760
4810     IF ERR=53 OR ERR=76 THEN 4830
4820     PRINT "ERROR NUMBER ";ERR;" DETECTED IN LINE ";ERL : END
4830     RESUME 750

```

OIL SAMPLING FROM RECOVERY TANKS

Samples are taken from the recovery tank sections using a stratified sample thief (See Figure A-6). Developed at OHMSETT, the thief is designed to simultaneously capture representative samples from a fluid column in three-inch (76.2 mm) segments (See Figure A-7). This eliminates the necessity of mixing the immiscible oil and water to obtain samples representative of the whole fluid column.

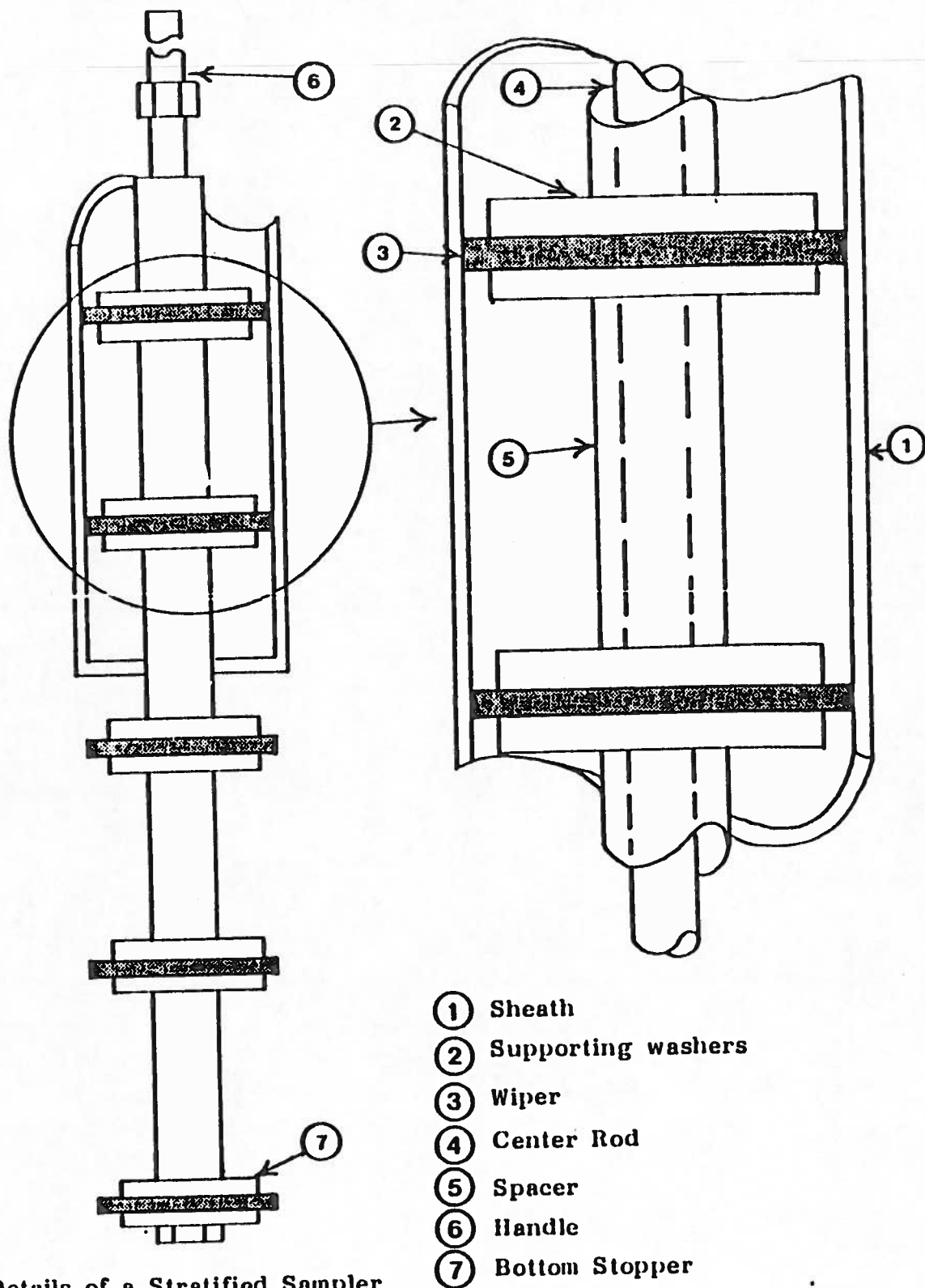
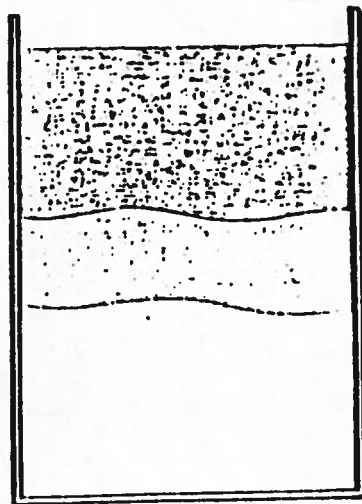
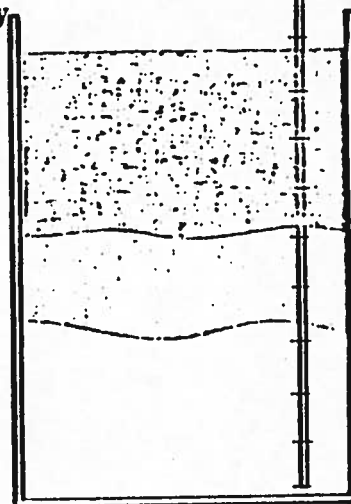


FIGURE A-6 - Details of a Stratified-sampler.

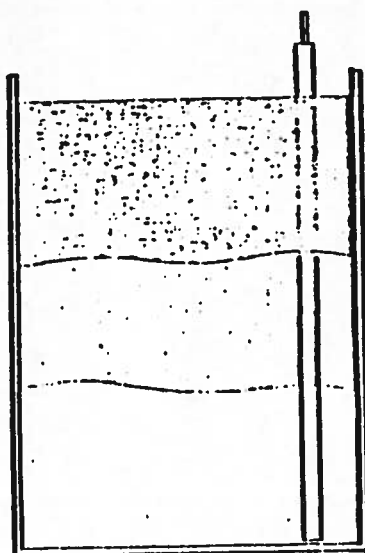


A. The sampler with the extension rod is placed in the Recovery tank.

B. After the sampler is in the tank the outer sheath is withdrawn to expose the center section



C. The outer sheath is slid down the center section, trapping the liquid.



D. The entire sampler is withdrawn from the tank with a representative sample enclosed.



FIGURE A-7 - How to use the stratified-sampler.

APPENDIX B

QUALITY ASSURANCE

QUALITY ASSURANCE PLAN FOR THE OFFSHORE BOOM AND SKIMMER TEST PROTOCOL

1.0 INTRODUCTION

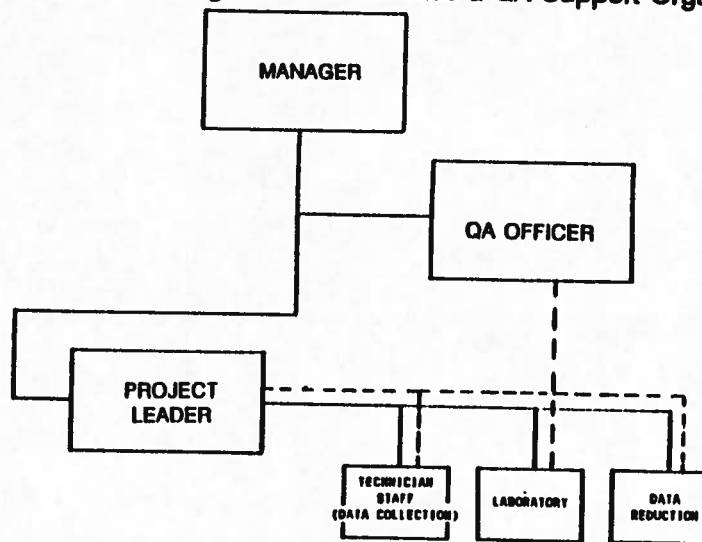
1.1 Introduction and Purpose

In measuring the boom and skimmer performance, the investigator is attempting to precisely measure processes that are being influenced by random events. Each instrument and each individual method can achieve high precision. However, the final data is often characterized by large standard deviations caused by waves. Long test durations reduce the standard deviations, but long tests, especially tank tests involving oil are often not considered practical. This Quality Assurance and Quality Control (QA/QC) Program has been designed to:

- Provide a management structure and report system that insures adherence to the accepted procedures for performing tasks described in the Offshore Boom and Skimmer Test Protocols.
- Obtain documented mechanical and analytical data of known data quality.

2.0 DATA QUALITY MANAGEMENT

Quality work depends upon the procedures activated for monitoring and control and support personnel responsible for each function. The following schematic outlines a QA Support Organization.



2.1 Project Manager

- Develop the specific work plan from the protocol.
- Have overall responsibility for meeting the protocol objectives.
- Have overall responsibility for assuring work force and technical expertise is provided.
- Be available to take action on any problem requiring additional management or technical

support.

- Review program reports, directives, QA plans, Health and Safety Plans, progress reports, and financial reports prior to release.
- Prepare periodic reports (overviews) and final reports as requested by MMS.
- Keep client informed of all aspects of the program activity including expenditures, progress, problems, and recommended solutions.
- Assure that all raw data, documentation, protocols, and reports are transferred to the archives at the close of each test program.

2.2 Test Director

- Ensure that the test equipment is assembled according to specifications and that it is working properly.
- Ensure that all chemicals, equipment, instrumentation, and standards needed for the test are acquired on schedule.
- Ensure that experimental work is performed in strict compliance with the selected test methods, the QA plan, and all the SOP's documented in the Work Plan.
- Direct all labor force operations from preparation and initiation to conclusion and demobilization.

2.3 QA Officer

The Quality Assurance Officer should be independent from the project team and will often report directly to a General Manager to maintain an objective function. In small organizations, this is not always possible. He will monitor and document the adherence of the facilities, equipment, methods, practices, records, and controls to the appropriate regulations. He will ensure that the data quality meets or exceeds the objectives. He will receive administrative and technical support, as needed, from the other members of the organization.

A summary of the QA Officer's responsibilities follows:

- Maintain copies of the master schedules, protocols (with up-dated amendments), program plans, and current standard operating procedures (SOP's).
- Audit the work project plan for adherence to the protocol.
- Inspect critical phases of work plan at the required frequency.
- Ensure that the work plan and standard operating procedures are followed by project personnel.
- Submit periodic reports to management.

- Review all interim and final reports.
- Issue standard operating procedures.
- Audit and keep records of calibration and traceability of standards.
- Audit data validation and analysis procedures.
- Oversee archiving operations.

2.4 Technicians

These individuals will rig the test hardware and carry out the work plan under direction of the test director.

3.0 DATA QUALITY OBJECTIVES

Every quantitative measurement contains sources of inaccuracy and imprecision, both of which have variable components. These have been recognized as Systematic and Random errors. Systematic errors affect the accuracy by giving a net bias to all of the measurements in the set. Random errors also make a noticeable contributions by reducing precision. Such variations arise due to instrument precision limits, sample size variability, residual sample heterogeneity, instrumental noise, and other technical limitations. Their effects are minimized by better engineering controls and a larger, well-defined database.

There are two basic indicators of measurement quality: precision and accuracy. When experimental measurements are first carried out by use of reliable working standards, excessive measurement variability provides a good reason to search for uncontrolled systematic errors. When evaluating results by validated methods, the final estimate of precision will usually rely on the assumption that all practicable steps have been taken to control (ie., suppress, eliminate, or compensate) the systematic errors. The remaining fluctuations are considered random and will determine the experimental precision.

3.1 Precision

Precision of a data set is measured as the standard deviation of that set using the calculation:

$$S = \frac{\sqrt{\sum (X_i - \bar{X})^2}}{N-1}$$

where:

S = Standard Deviation

X_i = Each member of a data set

X = Mean of the data set

N = Number of values in the data set

Initially, the averages of all the replicates is obtained and then, the standard deviation of each mean is calculated. This gives a measure of data dispersion for future comparisons. If any individual measurements in a 3 run set is more than ± 2 standard deviations away from the mean then another measurement must be performed for confirmation. The outlier in the set may then be discarded. In addition, the maximum number of replicates is always considered with the understanding that precision is directly proportional to the square root of that number; ie., 25 replicates result in a 5-fold improvement in precision and 100 replicates result in a 10-fold improvement in precision.

Precision is also expressed in terms of "repeatability" and "reproducibility". Duplicate tests are compared and a percent difference from the average value is calculated. Repeatability values are specified where large numbers of tests to determine standard deviations are not justified.

3.2 Accuracy

Accuracies of the measurements made in the protocol are supported by calibration procedures using traceable standards or lab-made standards following good laboratory procedures. Data accuracy objectives of some reported values are calculated from component measurement accuracies.

3.3 Completeness

The amount of data taken will depend on the specifications in the work plan. At a minimum, additional tests will be run only to replace outliers and examine nonconformance. Sufficient samples or tests will be done to ensure initial, middle and ending coverage.

3.4 Boom and Skimmer Test Protocol Quality Assurance Objectives.

See Table B-1 and Figure B-1.

TABLE B-1 - BOOM AND SKIMMER TEST PROTOCOL QUALITY ASSURANCE OBJECTIVES

MEASUREMENT	TEST	INSTRUMENT(S)	PRECISION	ACCURACY	CAL. STANDARD
Oil in Water	Oil and Grease, Total Recoverable (Infrared)	Infrared Spectrophotometer Quartz cells: 10, 50 and 100 mm Volumetric flask, 100 ml	Percent Standard Deviation in the 20 ml/liter range is 10%	Bias is +2.5%	Solvent extracts of blanks & "Spiked" dispersions of the test oil in tank water at: 1, 10 and 50 mg/liter oil Solutions of the test oil in CCl_4 at: 0, 10, 100, 500 mg/liter.
Water in Oil	Water and Sediment in Petroleum (Centrifuge)	Centrifuge with relative centrifugal force (rcf) equal to 500 at the fluid surface and 800 at the bottom. Centrifuge tubes, graduated.	Repeatability is dependent on the % water and sediment. At 7% water in oil: duplicate results by the same operator should be considered suspect if the standard deviation is greater than 0.4% water in oil.	There is no data available at present to determine accuracy.	Test oil with 1.5, and 10 % tank water blended in at high shear.
Specific Gravity	Hydrometer Method	Set of hydrometers Thermometer	Repeatability: Duplicate results by the same operator should be considered suspect if the results differ by more than 0.0006 grams/ml. Reproducibility: Results submitted by each of two laboratories should not be considered suspect unless the results differ by more than 0.0012 grams/ml.	+/- 0.1%	Distilled water Solutions of methyl alcohol and distilled water as needed over the range 0.79577 through 0.99727
Surface Tension & Interfacial Tension	Interfacial Tension of Oil Against Water by the Ring Method	Torsionwire tensiometer Platinum Ring	Repeatability: Same operator and apparatus, 2% of mean Reproducibility: Different operators and apparatus, 5% of mean	+/-1.5%	Distilled water Toluene NBS Class S fractional weights

TABLE B-1 - BOOM AND SKIMMER TEST PROTOCOL QUALITY ASSURANCE OBJECTIVES

MEASUREMENT	TEST	INSTRUMENT(S)	PRECISION	ACCURACY	CAL STANDARD
Viscosity	Viscosity - Brookfield (Rotary Spindle Viscometer)	Viscometer	Repeatability: +/-0.2% FS	+/- 1% Full Scale	Cannon-Fenske Viscometer(s) calibrated according to ASTM D 2162, sizes: 150, 200, 300, 350, and 400. OR Brookfield calibration standard fluids.
		Temperature Bath Thermometer			
Oil Flow Rate (Distribution Manifold)	Positive Displacement, Rotary Gear	Flow Meter, Totalizer			Measured 500 gallon container. AND Stopwatch, +/-0.01 seconds
		Stopwatch, +/-0.01 seconds			
Tow Speed	Over Ground Tow Speed, Tank Testing	Tow Drive coupled to a:	+/-0.05 knots	+/-0.05 knots	Surveyors tape AND Stopwatch +/-0.01 seconds OR Time Logger with external triggering.
		Rotating Light Chopper OR Magnetic Induction Sensor			
		Knot Meter or other flow sensor attached to the device or the tow vessel			
Relative Current Speed			Tank Testing: +/-0.05 knots Off-Shore: +/-0.2 knots	+/-0.05 knot	Tank Testing: Measuring Tape AND Stopwatch +/-0.01 seconds OR Time Logger with external triggering.
Wave Height and Period	Electronic Measurement of Wave Height and Period From a Fixed Platform OR Sonic Meter mounted above the water surface.	Resistance Wire, Pressure Gauge, Capacitance Probe mounted in the water	+/- 2.0 centimeters	96% responsiveness to a 1.0 meter/second depth change rate.	Off-Shore Testing: Calibrate in calm water pier-side as in tank OR calibrate in tank. Measuring Tape Stopwatch +/-0.01 seconds

TABLE B-1 - BOOM AND SKIMMER TEST PROTOCOL QUALITY ASSURANCE OBJECTIVES

MEASUREMENT	TEST	INSTRUMENT(S)	PRECISION	ACCURACY	CAL. STANDARD
Wave Height and Period (Continued)	Electronic Measurement of Wave Height and Period Using a Tethered Buoy	Wave buoy with accelerometers OR Spar-buoy with resistance wire	+/- 2.0 centimeters	98% responsiveness to a 1.0 meter/second depth change rate.	Measuring Tape Stopwatch +/-0.01 seconds
Slick Thickness	Calculated - average, for tank testing. No method has yet been identified for off-shore that can be relied on.	Oil Distribution Flow Meter Tow Speed Meter or knot meter Measuring tape Stopwatch +/-0.01 seconds	At 3.0mm slick thicknesses previous tank test data shows reproducibility to be +/-0.2mm	The accuracy of this measurement is dependent on the spreading/distribution of the oil over the water surface and the portion of the slick the device encounters. At 100% encounter accuracy, based on the individual accuracies of the component measurements, is +/- 5%	See standards for the individual instruments
Throughput Efficiency (% TE) The amount of oil skimmed from the water versus the amount encountered, expressed as a percent.	Calculated from: Total Oil Distribution Percent Encountered Recovered Oil	Visual estimate of percent encountered Oil Distribution Flow Meter Water in Oil analysis of recovered fluid. Depth Measurement of oil/water emulsion recovered	In calm water, throughput efficiency relative standard deviations for sets of six tests at 1.0 to 1.5 knots were 8.2 to 9.8%. In Harbor chop the relative standard deviations were 13.3 to 20.2%	+/- 5%	See standards for the individual instruments
Oil Recovery Rate (ORR)	Calculated from: Recovered Oil Duration of Recovery	Water in oil analysis of the recovered fluid Stopwatch Depth Measurement of oil/water emulsion recovered	No specific data available to support ORR precision. (See precision for %TE)	+/- 5%	See standards for the individual instruments

TABLE B-1 - BOOM AND SKIMMER TEST PROTOCOL QUALITY ASSURANCE OBJECTIVES

MEASUREMENT	TEST	INSTRUMENT(S)	PRECISION	ACCURACY	CAL STANDARD
Recovery Efficiency (%RE) The ratio of oil to water collected by the skimmer during the test.	Calculated from:	Water in oil analysis of the recovered fluid	No specific data available to support %RE precision. (See precision for %TE)	+/- 5%	See standards for the individual instruments
	Total volume of fluid collected	Depth Measurement of oil/water emulsion recovered.			
	Total volume of oil collected	Depth Measurement of total fluid recovered.			
Boom Wave Conformance	Dynamic Measurement of Boom Skirt Depth	Pressure Transmitters	Standard deviations of minimum depths measured for sets of five tests, each 4.3 minutes long, in wind waves and swell, range from 2 centimeters to 14 centimeters. Standard deviations for pressure transmitters at the sides of a catenary (P1 and P3) and at the apex P2 are presented in graphical form in Figure B-1.	Accuracy of boom wave conformance is dependent on maintaining a constant tow speed. In tank testing accuracy is calculated to be +/- 10% based on the combined instrumental accuracies. At sea tow tests are influenced by the coupling of boom and tow vessel(s) responses and test accuracy is reduced.	See standards for the individual instruments
		Wave Meter/Buoy			
		Tow Speed Meter/Knot Meter			

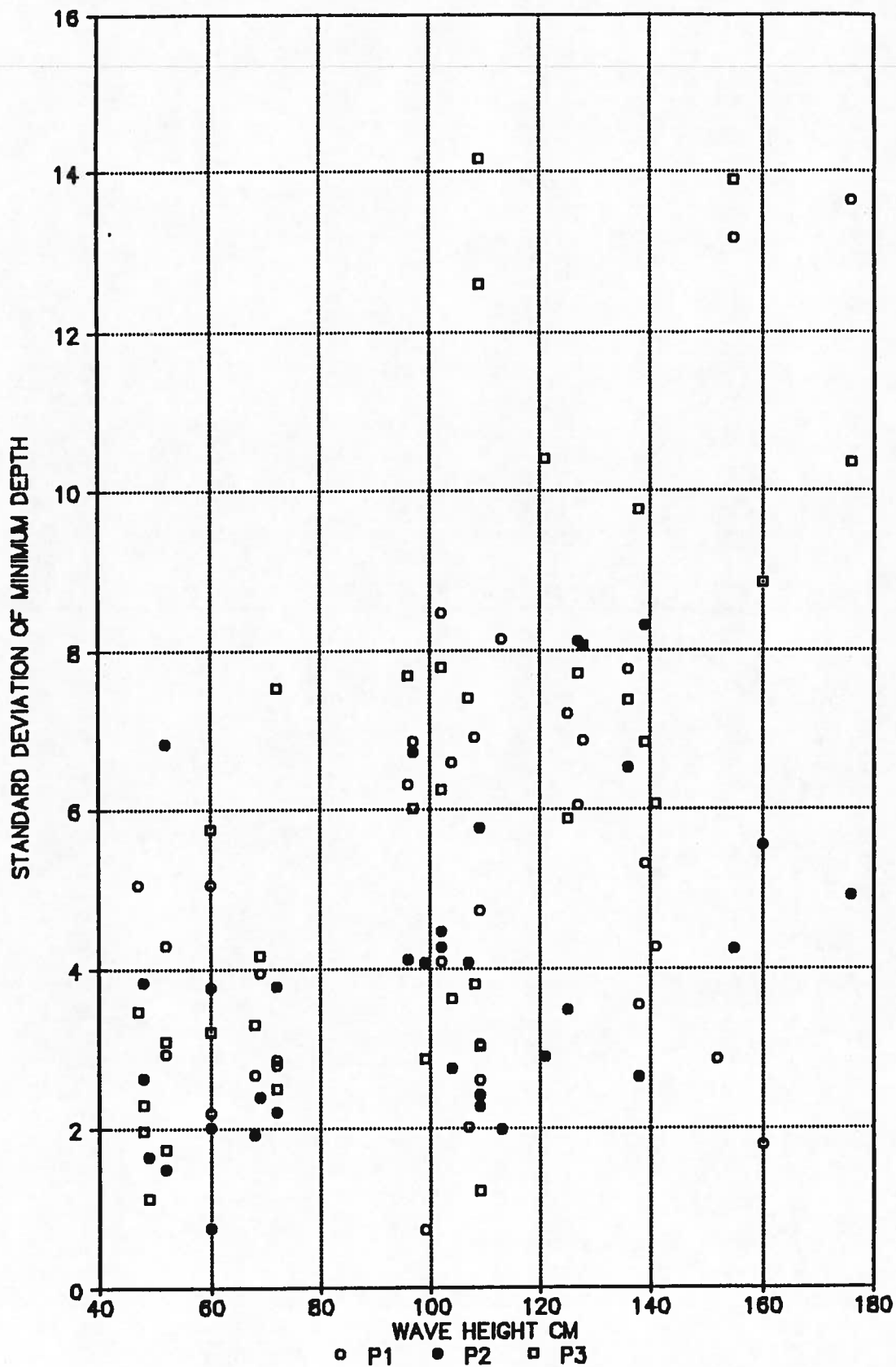


FIGURE B-1 - Standard deviations for minimum depth measurements. These values result from statistical analyses of off-shore boom evaluations. A three pressure transmitter array measured depths at the arms, P1 and P3, and at the apex P2.

4.0 CALIBRATION FREQUENCY

Each instrument should be calibrated prior to a test program and each day a two point calibration check should be performed. A two point check consists of a "zero" (baseline) reading and one other value in the top 25% of the measurement range of interest.

Some instruments such as pH meters, conductivity meters and surface tension balances are given a two point check with each sample.

5.0 PROCEDURES

5.1 The actual process of sampling just as the processing of the samples collected requires planning and quality control. Quality Assurance enters the picture with the selection of the sampling method, sample containers, preservation and holding times and covers the tracking of the samples or measurements from collection through analysis to data report. The key is sample identification to facilitate data traceability and summarization.

Samples are taken using a stratified sample thief. Developed at OHMSETT, and called the "Johnson Sampler", the thief is designed to simultaneously capture representative samples from a fluid column in 76.2 mm segments. This eliminates the necessity of mixing the immiscible oil and water to obtain samples representative of the whole fluid column. See Methods: Oil Sampling from Recovery Tanks.

5.2 Sample Custody - Oil and Water Analysis

A Chain of Custody Record has to be maintained for all of the samples generated on a daily basis. This form must follow the samples from generation through reporting of the analytical data. The Chain of Custody Record form duplicates most of the information recorded on the sample label and cross-referenced in the QA Log Book and goes further to document sample transfers. The sample collector fills in Part II and surrenders the sample and form to the QA Officer. At this point, the sample gets a QA reference number and enters the lab for analysis. The final step is the release of the data by the analyst, and the submission of the original Chain of Custody Record to the QA file and a copy with the written report.

5.3 See Appendix A for selected analytical methods used to obtain measurements necessary for the final data.

5.4 Data Collection

All data and measurements should have the following information recorded:

- Date of Analysis/Measurement
- Instrument Calibration/Standard Curves displaying:
 - Date/Time
 - Test performed and standard utilized
 - Initials of analyst
 - Test conditions/Instrumental Parameters

- Analytical data of oil or water must be recorded in a fixed-leaf notebook with the following information noted:
 - Initial of analyst
 - Test performed
 - Test conditions
 - Dilution factors
 - Visual observations (ie., significant differences)
 - Sample identity
 - Time sample was analyzed.
 - Person report sent to

6.0 DATA ANALYSIS, VALIDATION AND REPORTING

6.1 Data Sheets and Validation

The data sheets used for the collection of raw data must be prepared prior to testing to document all of the variables identified in each work plan. All critical data such as the dependent and independent variables listed in the test plan will be recorded on the data sheets each time a run is performed. Records will be kept in compliance with the guidelines given above. The accuracy of the data and calculations will be re-checked by the person performing the tests and by one other qualified person. In addition, the QA Officer will check the precision of the raw data and the equations or calculation methods used to calculate the dependent variables. The calibration, detection limits and recoveries will be evaluated. The data will be qualified for holding time exceedences (where applicable), blank contamination, spike recoveries, and detection levels.

6.2 Outliers

In order to obtain a complete data set, outliers must be identified in time to re-run specific tests

6.3 Reporting

Data sheets should be maintained during the rest program and at the end of each day reviewed for outliers and completeness. After validation, the data sheets will be submitted to the QA Officer who will maintain all raw data in notebooks. At the end of the study all data books will be bound and stored in a safe, accessible location.

7.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Spikes and conventional analytical standards must be analyzed.

7.1 Spike Sample Frequency and Procedure

Spike Sample Frequency and Procedure

Spike samples are to be submitted in a quantity equal to ten percent of the respective samples to be analyzed. This procedure is conducted as follows:

1. Known amounts of oil or water should be added to actual samples at concentrations where the precision of the method is satisfactory. It is suggested that amounts be

added to the low-concentration sample sufficient to double that concentration, and that an amount be added to one of the intermediate concentrations sufficient to bring the final concentration in the sample to approximately 75% of the upper limit of application of the method.

2. Two to five replicate determinations should be made at each concentration for instrumental analysis.
3. Accuracy should be reported as the percent recovery at the final concentration of the spiked sample. Percent recovery at each concentration should be the mean of the replicate results.
4. For convenience, samples selected to be spiked should be chosen from a set of samples previously analyzed for the desired spike constituent. This methodology will allow the contaminant level to be measured and the appropriate spike concentration to be added.

7.2 Duplicate Sample Frequency and Procedure

Duplicate samples should be obtained and considered in accord with the following stipulations:

1. They may be obtained from either of two sources:
 - Remaining portion of sample previously considered.
 - New sample obtained from same source, batch and sample point, mixed well and split.
2. The number of duplicates must depend on the analytical results but should be no less than 10% of the total number of samples tested.

In addition, the duplicate samples should be kept under the same conditions and run under the same parameters used for the original samples.

8.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

Normally the QA Officer conducts a performance audit on 10% of the runs. The first time, the QA officer will notify the operators that he will observe as they perform the tests and record the data. Subsequent observations will be unannounced.

As discussed in Section 10.0, all raw data sheets will be checked for accuracy and for compliance with test plan specifications.

9.0 PREVENTATIVE MAINTENANCE PROCEDURES AND SCHEDULES

A log book is maintained for every scientific instrument. The complete maintenance record is kept in each log book. Preventative maintenance checks and operations on a regular schedule will be performed. The QA officer will audit the records in order to insure that all necessary maintenance functions have been performed.

10.0 SPECIFIC ROUTINE PROCEDURES TO BE USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS OF SPECIFIC MEASUREMENT PARAMETERS

As discussed in Section 11.0, the QA plan for monitoring work in progress is to introduce spikes, surrogate samples and other standard sample analysis techniques. However, in studies where the former are not feasible the data quality is insured by checking for outliers.

11.0 CORRECTIVE ACTION

The QA officer conducts regular performance and data audits and relies on the test directors for corrective action deemed necessary. Both the performance audits and the data (record) audit require observation of the work in progress and annotation of the following information:

- Date
- Audit No.
- Auditor
- Project No.
- Phase of Study Inspected
- Test Material
- Procedure
- Procedural Step
- Findings
- Corrective Action Required
- Deadline for Correction
- Responsible Individuals Briefed
- Re-inspection Plan
- Corrective Action Taken
- Date Correction Finished
- Person Responsible for Correction

The completed statement is returned to the QA Officer for subsequent circulation to the Test Director, and Project Manager. The case is closed as long as the corrections follow the audit expeditiously and cause no noticeable effects on the data produced. Any discrepancy or trend outside the control limits will be closely examined and may require repeat of the questionable test.

12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

As described in Section 15.0 all audit reports are circulated to the Test Director and to the Project Manager. In addition the QA officer will write a periodic QA Report to the Project Manager, upon his request, to summarize the status of ongoing projects.